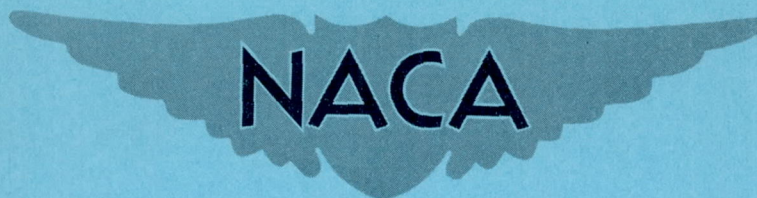


RM E54D27

NACA RM E54D27



RESEARCH MEMORANDUM

HIGH-TEMPERATURE LUBRICANTS AND BEARINGS FOR AIRCRAFT
TURBINE ENGINES

By NACA Subcommittee on Lubrication and Wear

Lewis Flight Propulsion Laboratory
Cleveland, Ohio

NATIONAL ADVISORY COMMITTEE
FOR AERONAUTICS
WASHINGTON

July 19, 1954
Declassified June 20, 1957

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RESEARCH MEMORANDUM

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SUMMARY

The problems, research status, and future possibilities for high-temperature lubricants and bearings for aircraft turbine engines were considered in detail by the NACA Subcommittee on Lubrication and Wear. Higher operating temperatures are inevitable for bearings and lubricants, although the extreme temperatures may occur for only short periods of time. The use of closed lubrication systems may permit operation with liquid lubricants at temperatures higher than now permissible. It may be necessary to develop separate lubricants for turbojet and turbo-prop engines. The use of more viscous fluids may allow a significant increase in the present limiting temperatures. Liquid lubricants may not be useful at the highest projected bearing temperatures (1000° F); solid and gaseous lubricants have promise for the higher bearing temperatures. Tool steels and cage materials are available that appear suitable for use in high-temperature rolling contact bearings, but considerable research is essential.

INTRODUCTION

A previous report (ref. 1) by the NACA Subcommittee on Lubrication and Wear, published in April 1951, was concerned with a review of current and anticipated lubrication problems in jet engines. At that time, bearing operating temperature did not exceed 350° F, and oil reservoir (bulk) temperature did not exceed 260° F. The report defined bearing temperature limits of 350° F for current (1950) interest and predicted limits of 500° and 750° F for the immediate and distant future. The predictions of higher temperature requirements were based on the trends, in jet engines, to higher compression ratios, higher operating temperatures, and higher mass flows to produce greater thrust. Some of the development and newer production engines are currently operating in the 350° to 500° F range.

At the time of the last report (ref. 1) it was stated that the diester fluids showed the most promise for the operation of bearings at higher temperatures. By virtue of a concentrated cooperative effort by industry, the military, and research agencies, the diester fluids have been developed into satisfactory jet-engine lubricants in a relatively short period of time. MIL-L-7808, the military specification for synthetic jet-engine lubricant, was issued in December 1951.

Bearing operating and bulk oil temperatures are higher today than they were 3 years ago and promise to go higher in the future. References 1 to 3, as well as consideration of extreme flight speeds, indicate that there are four general temperature ranges of interest as shown in table I.

TABLE I. - BEARING AND LUBRICANT TEMPERATURE TRENDS
FOR AIRCRAFT TURBOJET ENGINES^a

Temperature range	1	2	3	4
Engine status	Current	Development, new production	Future	Future
Maximum operating temperature, °F				
Bearing	< 350	500	750	1000
Bulk lubricant	250	300	400	----
Maximum bearing "soak-back" tem- perature, °F.	500	700	1000	----
Minimum tempera- ture for bear- ings and lubri- cant, °F ^b	-65	-65	-65 ^c	-65 ^c

^aSome of data from references 1 and 3.

^bTemperatures of -100° F have been encountered in service.

^cMay be necessary to sacrifice on low-temperature prop-
erties to achieve adequate high-temperature properties.

The tabulation shows that for current engines (temperature ranges 1 and 2), maximum bulk lubricant temperatures are 250° and 300° F; maximum bearing soak-back temperatures are 500° and 700° F (the temperatures attained by the bearings after the engine is shut down and coolant flow ceases while heat continues to flow into the bearings from hot parts of the rotor). In future engines, such as those for transonic and supersonic flight and particularly in engines for guided missiles, significant design objectives include maximum bearing operating temperatures of 750° and 1000° F (temperature ranges 3 and 4, table I). However, it should be noted that the problems involved in short-time engines (such as an expendable engine for a guided missile) may be considerably different from the problems for long-time engines (such as an engine to be used in piloted aircraft).

In order to obtain maximum performance from new engines, it is important that lubricants and bearings be developed to withstand higher temperatures. The purpose of this report is to define the problems, to present the latest available data on lubricants and bearings in order to evaluate our present research knowledge, and to indicate future possibilities. The body of the report contains summaries of papers presented by members of the NACA Subcommittee on Lubrication and Wear at the September 1953 meeting. Complete papers appear as appendixes A to G of this report.

TRENDS AFFECTING LUBRICANT AND BEARING REQUIREMENTS

High-Speed Aircraft Missions

High-speed aircraft missions are discussed in detail in appendix A. The military application of extreme flight speeds may be broken down into: (1) fighter application, and (2) bomber application, with long-range turbojet missiles falling into the bomber category. The maximum speed at which a turbojet power plant may be efficiently utilized lies above Mach 2.75, and may be as high as Mach 3.5 (appendix A). At these speeds, and at high altitudes, the effect of aerodynamic heating becomes a major problem. In the fighter application, the heat capacity of the system may still be effective because of relatively short flight duration, although temperatures much higher than now encountered will occur.

For the bomber application, the duration of flight at these extreme Mach numbers would be around two hours. This would allow time for the system to reach the high stagnation temperatures. The bomber case is the most critical in that fuel, normally used as the lubricant coolant, can no longer be used as it is at present. However, the relatively short time period associated with high Mach number flight makes it desirable to recognize the time-temperature concept in evaluating bearings and lubricants.

Engine Design Trends

Engine design trends are discussed in detail in appendixes B and C. Engines are being developed with greater thrusts and are being installed in aircraft designed for higher speeds. The higher ram temperature rise coupled with compressors that develop higher compression ratios results in much higher compressor-outlet temperatures. Reference 2 shows that compressor-discharge temperature for Mach 2 at 50,000 feet will be 660° F for a compressor pressure ratio of 5 and 1060° F for a pressure-ratio of 15. In addition, some current thinking is toward increases in turbine-inlet temperatures from 200° to 400° F over current practice of 1500° to 1600° F. A turbine-inlet temperature of 2000° to 2100° F is considered to be a practicable objective (appendix B). Another design trend which could affect lubricants and bearings is the use of preturbine fuel injection for thrust augmentation. Preturbine fuel injection moves the afterburner hot zone closer to the turbine bearing, which may aggravate the temperature problem.

Lubricant Cooling Problems

Lubricant cooling problems are discussed in detail in appendix C. High operating temperatures of the lubricant and bearings in turbine engines are due to the combined effect of high heat rejection to the lubrication system and limited cooling capacity. The sources of heat load in a turbojet engine include all main shaft bearings held in housings located in hot zones, the thrust bearing, accessory gearbox, pumping losses in fuel and lubricant pumps, constant-speed drive for alternating current power units, and aerodynamic heating effects. A breakdown of the heat rejected by each component for one particular engine of 10,000 pounds thrust is included in appendix C. Some of the data from this breakdown are shown in figure 1, which shows the effect of Mach number and altitude on engine heat load and oil-out temperature from the oil cooler. The total heat load for the engine of appendix C is considerably less than the heat load for another 10,000-pound thrust engine which has a rejection rate as high as 240,000 Btu per hour under severe conditions.

Although the heat load at the high-speed sea-level condition is greater than the load at high-altitude cruise, the sea-level condition has ten times the amount of fuel available for cooling than is available at altitudes (because of the reduced engine fuel consumption at altitude). Thus the minimum fuel-flow condition may become the limiting condition of operation.

The principal factor affecting bulk lubricant temperature will be the limitation on lubricant cooling. It would be possible to design a system to operate at lower temperatures by minimizing the heat that must be rejected to the lubricant and by increasing the cooling capacity of the system. However, most steps taken to reduce heat load and to increase cooling capacity result in added complexity and weight.

Cooling systems under consideration are fuel-to-oil, air-to-oil, and partially vaporized fuel-to-oil (appendix C). The fuel-to-oil cooler, in use at the present time, uses fuel on its way to the combustor as a coolant. The drawback to this system is the reduced fuel flow (cooling capacity) at high-altitude cruise conditions, and when descending from altitude. In addition, solid deposits of fuel decomposition products form when the fuel is subjected to high temperatures. The air-to-oil cooler is not feasible for high-speed application because of excessive drag and the ram-temperature-rise effect. Partly vaporized fuel-to-oil is attractive in that the latent heat of the fuel can be utilized for cooling. Although this system is especially attractive for combustors which utilize vaporized fuel, it offers problems in fuel metering and control.

Turboprop Lubricant Requirements

The lubricant requirements for turboprop engines are discussed in detail in appendix D. In the future, it appears that the lubricant requirements for the turbojet and the turboprop engines may be more difficult to reconcile with one lubricant than at present. The high-temperature requirements of the two engines are currently similar; but with supersonic flight speeds, turbojet operating temperatures will rise sharply whereas the turboprop will power high subsonic or low supersonic aircraft with attendant lower temperatures. The necessity of improved load-carrying ability of a given viscosity lubricant must be satisfied for the turboprop if low-temperature starting requirements and increased power ratings are to be realized. The present knowledge of lubricant compounding indicates that additives which increase load-carrying ability will limit the high-temperature stability of the lubricant. In addition, the search for better high-temperature lubricants is restricted if improved load-carrying ability, or even load capacity equivalent to the present MIL-L-7808, is a primary requisite. Therefore, separation of the two requirements appears advantageous if the desired rate of progress is to be attained.

HIGH-TEMPERATURE LUBRICANTS

Liquids

Because of the relatively low operating temperatures of most current engines (temperature range 1), petroleum lubricants meeting the MIL-O-6081A specification for grades 1010 and 1005 (10 and 5 centistokes viscosity at 100° F, respectively) are generally satisfactory. In temperature ranges 2, 3, and 4, however, where temperatures are appreciably higher, the petroleum lubricants do not have the required combination of properties such as low viscosity at -65° F for adequate pumpability and low volatility at high temperatures for low oil consumption. As a consequence, synthetic lubricants and other lubricant types (such as solids or gases) are necessary.

One of the very difficult problems with liquids is that of maintaining low volatility at high temperature while meeting the pumpability requirement for low viscosity at -65° F since, for any one class of lubricant, decrease in viscosity is generally accompanied by increase in volatility.

At temperatures above those at which liquid lubricants in open lubrication systems are practical, there are two general alternatives:

- (1) liquid lubricants in closed systems (with resultant limited oxygen availability), or
- (2) other lubricant types such as solids or gases.

Extensive thermal and oxidative stability research on various fluids is discussed in detail in appendix E.

Thermal stability. - Thermal stability tests were conducted in the absence of oxygen. At 500° F for a 20-hour period, esters, ethers, mineral oils, and silicones were fairly stable with the exception of esters prepared from secondary alcohol. Data on mineral oils, ethers, silicones, and silicates show that decomposition does not always result in an increased neutralization number. For these materials, change in viscosity, or the formation of insolubles, is more pertinent. Thermal stability tests at 680° to 750° F for 6 hours show naphthenic materials to undergo an appreciable loss in viscosity without significant volatility losses. On the other hand, aromatic oils show a slight increase in viscosity after being subjected to 700° F. As judged by volatile-product formation, Hercoflex-600, an ester made from primary alcohol linkages (pentaerythritol), appears to be reasonably stable up to 700° F. About 600° F appears to be the maximum thermal stability temperature for di(2-ethylhexyl) sebacate.

Oxidative stability. - In the presence of oxygen, different results are obtained. At temperatures above 450° to 500° F, the characteristic induction period of a well inhibited or stabilized oil no longer exists because the oxidation reaction is so rapid. Therefore, in studying oxidation at high temperatures, the important factor is the amount of oxygen assimilated in a given time by a given amount of oil, and the resulting effect on the properties of the lubricant. Tests conducted with di(2-ethylhexyl) sebacate at 500° F show phenothiazine to be effective in reducing the amount of oxygen taken in by the oil. Increasing the rate of air flow bubbled through the oil increases the amount of oxygen absorbed. Oxidation of a mineral oil in the presence of copper and steel results in a solid deposit. The esters are much less affected by the presence of metals at high temperature than are the mineral oils. The silicates undergo property changes after oxidation that are comparable to those for the most stable esters. The silicones as a class, however, are very stable (ref. 4 and appendix E). A blend of ester and silicone exhibits stability comparable to that of the ester alone. The effect of temperature level is illustrated in the following example: Di(2-ethylhexyl) sebacate, with 0.5 percent phenothiazine added, was stable in the presence of metal catalysts for 125 hours at 347° F. At 500° F the stable period was reduced to less than 1 hour.

The following considerations are important regarding lubrication at high temperature:

- (1) Restrict oxygen availability to the oil
- (2) Recognize the time-temperature concept
- (3) Continue to investigate materials such as silicones and esters of primary alcohols
- (4) Examine hydrocarbons in closed systems.

Lubrication properties. - From a lubrication standpoint only, a compounded diester, a phosphonate and a silicate appeared promising for high-temperature operation. (See appendix F.) The compounded diester showed effective boundary lubrication of steel-on-steel specimens to a bulk lubricant temperature of 500° F; at higher temperatures, incipient surface failure occurred. A phosphonate was a completely effective lubricant throughout the entire bulk lubricant temperature range to 572° F; at this temperature, however, the fluid refluxed violently, decomposed, and left a reddish-brown gel as a residue. The wear track on the friction specimens showed no damage and very slight wear, but the surface of the steel specimen appeared to be etched as if by chemical attack.

The tetra(2-ethylhexyl) silicate ester also appears promising for operation at high bulk lubricant temperatures. The silicate ester is, however, subject to oxidative decomposition at a bulk temperature of approximately 540° F. With the silicate ester, there was no visible evidence of etching of the steel specimens.

Testing the various components of the compounded diester indicates that tricresyl phosphate is the effective lubrication component. Studies at high sliding velocities indicate that, in general, fluids having a high critical sliding velocity have a high limiting temperature.

Some improvement in high-temperature lubrication properties of liquids can be obtained provided relaxation of the specification on low-temperature viscosity is permitted. For example, if it is allowable to use fluids of viscosities higher than the MIL-L-7808 specification value of 13,000 centistokes maximum at -65° F, improved high-temperature lubrication properties may be obtained. This point is illustrated in figure 6 of appendix F, which gives results obtained with a series of water-soluble polyalkylene glycol fluids that vary in viscosity from 8.9 to 762 centistokes at 100° F. The limiting temperature for incipient surface failure increased from 290° to 500° F with increase in viscosity.

The limiting temperatures at which lubrication failure occurred are not necessarily the bulk lubricant temperatures to which the lubricants can be utilized in practical bearing lubrication. Factors other than lubrication failure may become the limitation; for example, chemical effects such as decomposition, corrosion, and oxidation may limit the bulk temperature, to which the lubricant can be used, to values lower than those indicated in appendix F. Since the chemical effects are time-temperature dependent, however, it may be possible to use these lubricants up to the lubrication failure temperature for short-time applications such as expendable engines for guided missiles. For long-time operation, the use of these fluids will probably be limited to temperatures lower than those for lubrication failure.

The maximum allowable bearing temperatures may, however, be substantially above the bulk oil temperature limits indicated herein. In bearing lubrication,

the oil is normally supplied at low "oil-in" temperatures, and the oil residence (dwell) time may not be sufficient so that high bulk oil temperatures are obtained. In some full-scale bearing experiments with low oil-in temperatures, adequate lubrication has been obtained with bearing temperatures approximately 200° F higher than the temperature reported herein for incipient surface failure with the same lubricant.

Wear data (appendix E) over the temperature range 165° to 400° F indicate that tricresyl phosphate is an effective antiwear agent over the entire temperature range. Tests with a production-model Vickers piston pump show that currently available pumps can be operated successfully with relatively low-viscosity, high-volatility fluids provided good lubricity characteristics are built into the fluid. A silicate fluid shows wear and E. P. properties similar to nonadditive mineral oil and ester fluids. Fluids of this lubricity level have been successfully used as hydraulic fluids. The silicones show very poor lubricity properties. The addition of up to 50 percent by weight of ester to the silicone improves wear characteristics.

Solids and Gases

Liquids may be inherently temperature-limited because of thermal or oxidative instability; however, solid or gaseous lubricants have promise for the extreme high-temperature applications (refs. 5 to 7). Solids can be obtained which are either stable at the elevated temperatures or whose instability does not adversely affect the frictional properties of the surfaces to be lubricated. Similarly, gases (such as air) are extremely stable at elevated temperatures.

Solids may be used for lubrication in essentially two manners: (a) by including the solid as a minor constituent of the material to be lubricated, and (b) by introducing the solid with an "air-mist" system similar to that described in reference 5 or mixed with a volatile fluid carrier. Detailed discussions of both methods are included in appendix F. It is shown in reference 5 that it may be possible to effectively lubricate conventional rolling contact bearings with a solid (MoS_2) either at high temperatures (to 1000° F) or at high speeds (to DN values of 1,000,000).

As discussed in appendix F, gases may also be utilized as lubricants at high temperatures. Some preliminary results reporting the use of a reducing atmosphere (consisting of an air-hydrocarbon mixture) to lubricate small rolling contact bearings at 10,000 rpm are reported in reference 6. These results showed that successful operation could be obtained at temperatures of 600° F for several 10-hour periods. Other gases, such as air, may be employed provided bearings are designed for this fluid. The studies of reference 7 show that the air bearing (which is essentially unaffected by temperature) has possibilities for supporting load in a relatively stable manner at temperatures of 1000° F.

Temperature Ranges for Various Lubricant Types

A summary of the possible lubricants for operation over the four temperature ranges of table I is shown in table II. As shown, petroleum lubricants of the MIL-O-6081A type are suitable for temperature range 1; synthetic lubricants (presently, diesters) of the MIL-L-7808 type are suitable for temperature range 2; solids, gases, or liquids (possible future synthetics) may be suitable for temperature range 3; and, in all probability, only solids and gases will be suitable for the extreme conditions of temperature range 4. As indicated in table II, additional

TABLE II. - TYPICAL LUBRICANTS AND BEARING MATERIALS FOR VARIOUS TEMPERATURE RANGES

Temperature range	1	2	3	4
Maximum operating temperature, °F Bearing Bulk lubricant	<350 250	500 300	750 400	1000 ----
Minimum temperature, °F	-65	-65	-65	-65
Possible lubricants	Liquid-petroleums	Liquid-diesters + other synthetics	Liquids in open or closed systems	Solids or gases
Research required on lubricants	None	High-temperature stability and load-carrying capacity (for turboprops)	High-temperature stability and lubrication in open or closed systems	
Possible rolling contact bearing materials (A) For races and rolling elements	SAE 52100	Tool steels SAE 52100 (interim material)		
(B) For cages	Ag-plated bronze	Nickel alloys Nodular iron Ag-plated bronze (interim material)		
Research required on bearing materials (A) For races and rolling element materials	None	Fatigue research		
(B) For cages	None	Friction and wear research		

research is required on high-temperature stability and high-temperature lubricating effectiveness of lubricants for temperature ranges 2, 3, and 4. The use of closed systems (excluding oxygen) may make the use of liquid lubricants possible at temperatures higher than those of range 2.

HIGH-TEMPERATURE BEARINGS

Bearing Materials

Materials for races and rolling elements. - The physical properties and requirements of bearing materials for operation at high temperatures have been discussed in reference 2. The most important required properties are as follows:

- (1) Minimum hardness of Rockwell C-58
- (2) Dimensional stability at operating temperature
- (3) Minimum critical alloying elements (particularly tungsten)
- (4) Corrosion resistance at operating temperature and at room temperature
- (5) Reasonable heat-treatment and grinding characteristics
- (6) Ready availability from several sources

Based on these requirements, the molybdenum tool steels appear to show considerable promise as high-temperature bearing materials. Molybdenum tool steels have good hot hardness and dimensional stability at elevated temperature, although they are more difficult to grind than SAE 52100 steel (Wellons, ref. 2). The general discussions of the papers presented in this report as appendixes emphasized the importance of fatigue and the lack of data on fatigue lives of the tool steels at high loads, high DN values, and high temperatures.

Although tool steels will undoubtedly be required for temperature ranges 3 and 4 of table I, some thought has been given (appendix G) to using SAE 52100 steel stabilized for use at temperatures above the present limit of 350° F. Draw temperature must take into account both the required degree of dimensional stability and the minimum acceptable hardness.

A stabilizing heat treatment for SAE 52100 which specifies a 4-hour draw at 235° to 240° C (approx. 460° F) resulted in Rockwell hardness values of C-58 to C-61. Although this treatment assures adequate dimensional stability for operating temperatures up to 350° F, recent experience with high soak-back temperatures after engine shutdown demonstrated that 4 hours was not sufficient for complete stabilization. An 8-hour cycle at 250° C (482° F) for SKF No. 1 steel or 240° C (464° F) for SAE 52100 is desirable for the complete dimensional stabilization of bearings which are intended for operation where the temperatures may reach 400° F or higher.

Cage materials. - As shown in reference 2, the requirements for cage materials are as follows:

- (1) Adequate strength
- (2) Corrosion resistance
- (3) Compatibility with race materials
- (4) Thermal expansion coefficient approaching that for the race material

Of these requirements, that of compatibility (low friction and wear) is probably the most important and possibly the most difficult to obtain.

Friction and wear data have been obtained for a number of possible cage materials sliding against M-10 tool steel at temperatures up to 1000° F (appendix F). Conventional (Alcop) bronze, a presently used cage material, showed a continuous increase in friction with higher temperatures; wear first decreased, then increased with temperature. Temperature had no effect on iron-silicon bronze to 600° F. At higher temperatures both friction and wear increased. Friction for nodular iron decreased with increased temperature but wear increased slightly. However, wear characteristics of the nodular iron were better than for the bronze. In general, the friction coefficient for nickel alloys decreased with increasing temperature. Wear for the nickel alloys was approximately 1/2 and 1/10 that of iron-silicon bronze at 600° F and 1000° F, respectively. Nickel alloys can be pretreated to give low wear at room temperatures (fig. 11, appendix F).

Temperature Ranges for Various Bearing Materials

A summary of the possible bearing materials (both for rolling contact and for cages) for operation over the four temperature ranges of table I is shown in table II. As shown, the present race and rolling element material (SAE 52100) and the present cage material (silver-plated bronze) are suitable for temperature range 1. Likewise, the present bearing materials may be used, until more information is available on other materials, for temperature range 2; this is, however, only an interim measure, and the molybdenum tool steels and other cage materials (such as nodular iron and nickel alloys) may be used in the near future. For temperature ranges 3 and 4, the rolling contact bearings will undoubtedly employ tool steels for races and rolling elements, and nickel alloys, nodular iron, or other special materials for the cages. Very little is known, however, about the performance of these materials under these conditions. Much additional research is required on fatigue as well as on friction and wear in temperature ranges 2, 3, and 4. Full-scale bearing tests under simulated operating conditions must be employed as the final check of new materials or designs.

CONCLUDING REMARKS

The following remarks summarize the important points described in the discussion of high-temperature lubricants and bearing problems.

1. The greater heat loads imposed by high-performance engines and practical limitation on cooling will result in increased operating temperatures for bearings and lubricants.

2. The time-temperature concept and the use of closed lubrication systems become important considerations in obtaining solutions to high-temperature lubrication problems.

3. The attempt to develop one lubricant suitable for both turbojet and turbo-prop engine types may have to be abandoned as being unduly restrictive.

4. The use of more viscous lubricants may allow operating at higher temperatures but may require relaxation of the low-temperature pumpability requirements.

5. Solids and gases have promise as possible lubricants for bearing temperatures as high as 1000° F.

6. Tool steels appear promising for application to high-temperature rolling contact bearings. Preliminary studies indicate that friction and wear properties of tool steels are comparable with those of SAE 52100. Considerable fatigue data are required on the tool steels.

7. On the basis of bench studies of friction and wear properties only, certain materials appear to have promise for possible use in cages of rolling contact bearings for operation at temperatures as high as 1000° F.

Lewis Flight Propulsion Laboratory
National Advisory Committee for Aeronautics
Cleveland, Ohio, May 11, 1954.

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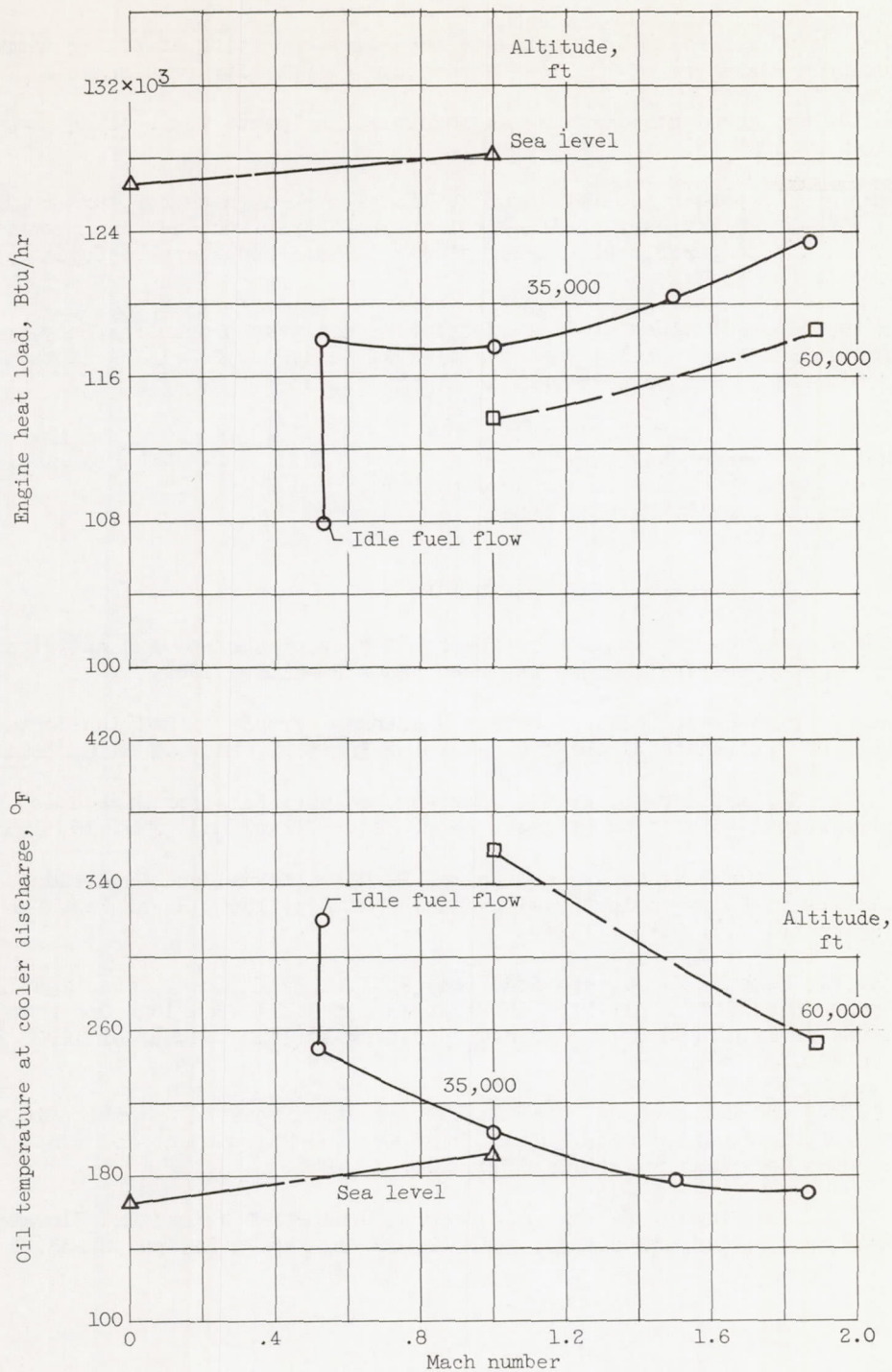


Figure 1. - Effect of flight speed and altitude on engine heat load and oil temperature at cooler discharge. (Data from appendix C.) (All points at military engine rating except as noted.)

APPENDIX A

HIGH-SPEED AIRCRAFT MISSIONS

By C. M. Michaels
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In this review of high-temperature lubricants and bearings for aircraft turbines, it was felt that a discussion of some of the contemplated high-speed aircraft missions is appropriate.

Two considerations of operating conditions are important: first, that high speed creates problems; and second, that the high-speed conditions occur for relatively short periods of time. The duration of missions may be only a few hours, although in these few hours great distances are covered. It is believed that these high speeds and their relatively short duration will dictate revisions of some concepts in the field of lubrication.

The military use of extreme speeds may be separated into two categories which are typified by a fighter application and a bomber application. The guided missile, which would use a turbojet engine, is considered to fall within the bomber category; since only a limited amount of maneuvering should be expected in its flight path, the engine would be operated at constant speed, and the duration of flight would approach that of the bomber. Extreme speeds, which may be defined as the maximum speed for which a turbojet engine may be utilized, are considered by various authorities to be above a Mach number of 2.75 and perhaps as high as 3.5. Within the fighter category, the following two missions may be considered: First, a somewhat normal take-off, climb, and cruise, with extreme speeds which would apply in the combat area for a period of the order of 15 minutes; and second, the use of essentially the same aircraft but with all flight phases conducted at maximum speeds. In the practical case, this length of time is limited to approximately 30 minutes. In the fighter application, the heat inertia of the system is probably still effective, although temperatures much higher than are normally encountered will occur.

For the bomber application, in some instances, the duration of flight at these extreme Mach numbers would be of the order of 2 hours. This period of time would normally allow the temperatures associated with these high-speed flights to stabilize throughout the system. The bomber case is therefore considered to be the most difficult, based solely on the temperature problems. It is obvious that bearing temperatures and oil temperatures tend to increase significantly. The high temperature of the available heat sink makes the problem of maintaining the oil and bearing temperatures within limits extremely difficult.

It is desirable at this point to discuss possible changes in concept regarding the physical properties of the lubricant for high-speed applications. The temperature limits of the lubricant are defined by fairly conventional tests, many of which involve time. It has been indicated previously that the time during which the engine is operated at high speed, and therefore the duration of high-temperature conditions, is limited. If the time elements used in defining the physical properties of the lubricant could be reduced, the allowable lubricant temperature could be increased. Similar considerations apply to bearings. Since very few data have been obtained which apply to the operation of the bearings at extreme temperatures, any known data should be extrapolated with caution. However, it is believed that bearings of the rolling contact type can be made usable at temperatures of approximately 700° F, although the goals for such elements should be much higher.

Another important consideration for high-temperature lubricants and bearings is the cooling problem. As the first step toward a solution, the bearings should be mounted in such a way as to minimize the amount of heat entering the bearing; however, additional means of cooling must be provided. One available heat sink is the engine fuel. Many current engines are equipped with fuel-to-oil heat exchangers in which the heat rejected to the oil is transferred to the fuel. However, this heat sink will also be required to cool other components such as electronic equipment, aircraft skin, and hydraulic fluid. It is also possible to allocate some of the fuel exclusively for cooling; this fuel could be evaporated and discharged overboard or, if possible, added to the afterburner. This process of adding the fuel vapor to the afterburner is as yet unrealized, although the associated problems do not appear to be insurmountable. When this possibility for a simple refrigeration mechanism is considered, it is immediately apparent that water would be a better medium, although it would complicate the system to some extent. The only alternative to such a consideration would be the use of a mechanical refrigeration system. The heat rejection to the oil of one of the latest types of production engines is about 15 tons of refrigeration (180,000 Btu/hr), which would require a large refrigeration installation. Since the evaporation of fuel would require approximately 24 pounds of fuel per hour per ton of refrigeration (2000 Btu/min), it becomes evident that this scheme would provide both simplicity and weight savings in the short-time usage. The point at which the method will no longer be advantageous has not been determined.

In summary, it may be stated that maintaining adequate lubrication and bearing systems for extended periods is believed to be one of the limiting problems in the application of turbojet engines to high-speed flight. The problem may be divided into the following phases:

- (1) Lubricants which are applicable for high-temperature use
- (2) Bearings which can withstand high temperatures
- (3) Elimination of the rejected heat from the over-all aircraft system.

APPENDIX B

ENGINE DESIGN TRENDS AFFECTING LUBRICANTS AND BEARINGS

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The general trends in aircraft turbine engine design point toward problems with lubrication and bearings. As is generally known, engines are being developed with greater thrusts utilizing higher compression ratios and are being installed in aircraft designed for higher speeds. The higher ram-temperature rise with higher speeds combined with higher compression ratio results in much higher compressor-outlet temperatures. The direct effect of this temperature increase on bearings and lubricants will depend to a certain extent on the design compromises chosen by the engine designer, but will almost inevitably result in some added heat rejection to the lubricating oil.

Changes in turbine temperatures and turbine temperature control are also in prospect and may affect heat rejection to bearings in the turbine end of the engine. Current thinking in the Bureau of Aeronautics contemplates increases in turbine-inlet temperatures of 200° to 400° F over current designs. These temperature increases may be achieved through the use of new metal alloys and turbine blade cooling. A turbine-inlet temperature of 2000° to 2100° F is considered to be a practicable short-range objective. The over-all effect of increased turbine temperatures together with blade cooling cannot be assessed directly in terms of total heat rejection to the oil or in terms of bearing operating, or soak-back, temperatures. Again this will depend on the designer's choice of compromises.

The principal factor affecting bulk lubricant temperature will be the limitation on lubricant cooling. Because of the drag induced by an air cooler and its decreased cooling capacity at high speeds, most new turbojet engines are being designed with fuel-oil coolers. Under certain conditions (principally when descending from altitude) the fuel flow is limited and oil temperatures will increase. New designs will generally provide for cooled-oil temperatures in the neighborhood of 300° to 325° F under this limiting condition. This is about the upper limit for the current synthetic oils; above this temperature, oxidation seriously reduces the life of the oil. If oils are developed which will withstand higher temperatures, fuel stability will then become a limiting factor at cooled-oil temperatures somewhere between 300° and 400° F depending on restrictions that are possible on fuel type.

Current estimates would indicate an upper limit on flight speed of approximately Mach 2 for Navy aircraft during the next 10-year period. With consideration of the altitudes at which this maximum Mach number will be experienced, it is possible to predict ram air temperatures not in excess of 350° F. The development of a lubricant which would have bulk stability at temperatures of 450° to 500° F would make it possible to utilize air-oil coolers either to replace or to supplement fuel-oil coolers in the event that the heat capacity of the fuel stream becomes severely limiting.

A current trend in Navy thinking on turbojet engine development may have some significance insofar as bearing loads and speeds are concerned. This thinking envisages the development of small, light engines which may be used in multiple installations. These engines would be no larger than the present J46 and would employ pressure ratios of perhaps 7 to 8. It would appear that bearing loads and speeds would not change appreciably from present designs.

Another design trend which might affect lubricants and bearings is the trend toward preturbine fuel injection for thrust augmentation. Preturbine fuel injection would move the afterburning hot zone closer to the turbine and might result in temperature increases or higher heat inputs into rear turbine bearings during periods of afterburning.

In summary, current development trends toward engines with higher pressure ratios and higher turbine temperatures may result in higher rates of heat rejection to the lubricant and higher bearing temperatures. As a modifying factor, consideration is being given to the development of relatively small engines which will not necessarily utilize pressure ratios above 8. No matter how the heat rejection rate to the lubricant is affected, the problem of heat rejection from the oil will become increasingly serious with higher-speed aircraft.

APPENDIX C

PROBLEMS ENCOUNTERED AT HIGH TEMPERATURES IN

LUBRICATION SYSTEMS OF TURBINE ENGINES

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The high operating temperatures of the lubricant in turbine engines are due to the combined effect of high heat rejection to the lubrication system and limited cooling capacity available in the lubricant cooling system. It is possible to design the lubrication system to operate at any reasonable temperature by minimizing the heat rejection to the lubricant and by providing cooling capacity to give the designed temperature. However, most steps which are taken to minimize heat rejection to the lubricant add complexity and weight and sometimes impair engine performance, and the steps necessary to provide cooling capacity also add weight and sometimes complexity; the engine designer therefore usually finds that the best design compromise is that which allows the lubricant to operate at the highest practical temperature. This is not always true, however, and each design should be completely studied.

The engine designer takes advantage of every improvement in over-all lubricant and material development which allows him to operate at elevated lubricant temperatures because this will result in reduced weight and improved performance.

The source of high heat load in a turbojet engine will now be considered. Table I shows a heat load and heat-balance summary for an engine of 10,000 pounds thrust. This heat load comes from the following main sources:

(a) All main shaft bearings which are held in housings located in the hot zones of the engine absorb heat. Heat is conducted into the lubrication system through the walls of these housings, depending on the exposed surface area to the hot gases and the velocity of the gases and fluids on either side. By minimizing these areas and surface velocities, heat rejection to the lubricant from this source can be held to a low level. The tendency of increased heat load from this source in future engines is apparent, because of the trend towards higher gas temperatures, but judicious design and proper insulation will help to compensate for this trend.

(b) Heavy load on the engine thrust bearing is a large source of heat load. The thrust on the engine thrust bearing is usually the small difference between two large numbers - the forward thrust of the compressor balanced against the rearward thrust of the turbine. A weight study should determine the relative advisability between the use of a large thrust bearing, or the use of thrust balancing by air pressure or by adjustments in the location of the various engine air seals. However, it should be remembered that the higher thrust loads also increase heat rejection which must be dissipated. To get rid of the heat requires additional cooling capacity, which means additional weight, and sometimes additional drag. It is advisable for aerodynamic designers to take a second look whenever the design results in a thrust bearing load that is on the high side. Some of the most efficient engines in operation today have compressor and turbine design combinations which result in a reasonably close balance in forces.

For a given altitude, increased flight speeds cause increased thrust bearing loads, as the thrust goes up from static sea-level rating almost directly with ram-pressure ratio. A high static thrust load is therefore multiplied at higher flight speeds, resulting in additional heat to be rejected to the lubricant.

(c) The accessory gearbox heat load consists of the sum of a fixed heat load that is a function of rotative speed only and a variable heat load which increases with the horsepower requirements of the gearbox. However, the surfaces of the gearbox contribute somewhat to the cooling of the lubricant, even at relatively high flight speeds.

(d) Pumping losses in the lubrication system account for an appreciable amount of the heat load, but the degree of effect varies considerably with engine configuration. For example, engines with a very simple oil system which have low oil flows and do not use the lubrication system for engine control have a very low heat load from pumping. However, the present trend to afterburning engines, with the need for exhaust-nozzle control, and the trend towards high-pressure-ratio compressors with the need for variable engine geometry necessitate some means for actuation of these parts. In most engines this is done by using engine oil and a high-pressure pump. To minimize the heat load, variable-displacement pumps sometimes are used.

The greatest pumping loss is that which occurs in the fuel pumping system due to the fixed-displacement fuel pumps used on American engines, and due to the need for high fuel flows for low-altitude, high-speed flight operation. Since higher-altitude operation requires considerably lower fuel flows, it is necessary to bypass the excess fuel. This extra work of pumping is converted directly into heat in the fuel and has the same effect on oil temperature as heat load anywhere in the system. Variable-displacement fuel pumps would correct this situation, but to date engine designers have not found this practical. The use of multiple fixed-displacement pumps which run unloaded when not needed is one method presently used to solve this problem.

(e) Another piece of hydraulic equipment now being included in the engine oil system on advanced engines is an integral constant-speed drive unit. It is made integral with the engine because considerable weight can be saved on the airframe by elimination of separate reservoir, cooler, pumps, and piping systems. The purpose of this is to drive an alternating-current power system for operation of aircraft electronic equipment. Such systems have been found to greatly reduce aircraft weight because of the use of higher voltages, and consequent reduction in weight of wiring, the elimination of inverters, and so forth. The constant-speed drive units, however, have added a heat load to the engine oil system that is roughly half the basic engine heat load.

(f) High-speed flight adds to the severity of the oil temperature problem because the ambient temperature in which the engine operates is increased. Although this is generally credited as the reason for the higher operating temperature of the lubricant, and the resulting problems, there are compensating factors during high-speed flight which tend to reduce oil temperature. As a result, there is only a slight increase in operating temperature of the lubricant during high-speed flight. For example, during high-speed flight the fuel available to the fuel-oil cooler is greatly increased, with the result that the actual maximum oil temperature is only slightly increased.

In the following paragraphs, engine cooling systems are discussed:

(a) Some engines have no cooling system whatever and rely only on the heat rejection from the various surfaces to supply a heat balance. This can be done,

providing the design is such that the total heat rejection to the engine oil system is low. Such an engine has the advantage that the weight of the oil cooler and associated piping is saved.

(b) Some engines provide a fuel-to-oil heat exchanger in which the fuel on the way to the engine combustor is heated by the engine lubricant on its way to the engine bearings. This is a very effective and reasonably light means of cooling the lubricant. The weight of such coolers is about 15 pounds and there is no appreciable drag penalty.

(c) Some engines require that the aircraft supply an air-to-oil heat exchanger to cool the engine lubricant. Such coolers are heavy and cause severe drag losses to the aircraft. This type of cooler is not practical for high-speed flight, since it would be effective only as a heater.

(d) For some engines with very high heat rejection, the maximum safe lubricant operating temperature would be exceeded when only a fuel-oil heat exchanger is used, especially in the range of operation where fuel flows are low, but where heat rejection is high; in such a case, a combination heat exchanger is used, where air-oil heat exchanger is automatically cut in by a thermostatic valve. Under all other conditions (where fuel flows are higher), the fuel-oil heat exchanger is sufficient. This system is a rather heavy one and should be avoided if possible.

(e) An effective means of obtaining added heat dissipation has been tried on some engines; this method involves an oil cooler in which some of the fuel is permitted to vaporize; thus, added cooling capacity is obtained from the latent heat of vaporization of the fuel. This system can be very readily applied in engines which have vaporizing combustion chambers.

(f) Refrigeration equipment in the oil system has not yet been found necessary by any engine manufacturer. The reason for this is simply that the engine designer has not yet done all he can do to limit heat rejection, or has not yet taken maximum advantage of the elevated temperature to which present lubricants and seal materials will go. No doubt by the time present limits are reached, other lubricants capable of operating at higher temperatures will be available, as will higher-temperature sealing means.

To sum up, the problems which are listed need not be problems at all except that the designer finds it better to solve them than to add the extra complexity and weight to the engine that would allow him to operate at lower temperatures.

These problems are listed in various categories. The problems which are directly associated with operation of the lubricant fluid at high temperature will be considered first.

The lubricant has a tendency to lose its ability to lubricate with increased temperature. This is primarily due to the fact that the lubricating characteristics of a given fluid decrease with viscosity. As a result, new fluids with higher viscosity indexes must be found. The use of the diester-base fluid MIL-L-7808 was a big step forward, but other fluids with improved properties are being developed. Generally speaking, however, this drop off in lubricating ability at elevated temperatures has not been a primary problem in turbojet engines to date, as the development of the fluid has kept up with the increase in operating temperatures from this point of view. However, from the point of view of stability, the diester-base fluids have not kept pace with operating temperatures. At the present time it is necessary to change the engine lubricating fluid periodically - more frequently on engines operating at very high temperatures - in order to prevent the fluid

neutralization number from going too high, and to prevent the formation of objectionable sludge. A more stable lubricating fluid than MIL-L-7808 is very necessary. The present rate of sludge formation is severe enough to cause clogging of filters (and oil jets downstream of the filter after the filter relief valve begins to bypass). Filters must be changed quite often. Operation of MIL-L-7808 well beyond the specification limit for neutralization number has had no apparent detrimental effects with regard to corrosion of the parts. In general, the operation of MIL-L-7808 fluid in the 300° to 400° F range has had no detrimental effect except for the oil sludging problems. It is believed that this problem could be eliminated without change to the fluid if the continuous source of new oxygen were eliminated. With this in mind experiments are now being run on closed lubrication systems in which the vented air is not exhausted overboard, but is sent back to the bearing housings and recirculated. To accomplish this, however, added complication is necessary to the engine design. Positive oil seals must be used which are capable of operation in an ambient temperature of 1000° F. Such seals are available but must be developed for each specific application.

The closed lubrication system automatically eliminates another problem, excess oil consumption due to high volatility of the lubricating fluid and loss of this vapor through the oil-system vent. At the present time, engine oil consumption increases considerably under "hot oil" condition because of loss of vapor out the vent.

The toxicity problem of the lubricant is present at extremely high operating temperatures for the diester-base fluids. Above 600° F the fluid breaks down to form some aldehydes which cause nausea when breathed. This problem is not readily apparent at present operating temperatures except where the fluid leaks into the compressor flow path and moves back to a point where the temperature is high and where bleed air is taken for cabin pressurization. At temperatures above 600° F, this fluid does break down and if sufficient leakage has been present, the bleed air may be sufficiently contaminated to cause nausea among the crew of the aircraft.

Sealing problems at high temperatures are a natural thought in view of the previous discussion. To keep oil consumption low, and especially to prevent contamination of bleed air, positive reliable seals should be used at all points forward of the air bleed ports. These seals consist of such components as carbon, steel, springs, rubber, silicone rubber, bellows, and piston rings. For temperatures up to 300° F, some rubber is used; up to 450° F silicone rubber is used; and at still higher temperatures, all-metal construction is used. As mentioned before, seals are available to operate in ambient temperatures up to 1000° F; however, each seal application is a development problem in itself, especially those in which no rubber may be used. It is believed that the shaft sealing problem can be handled by the engine designer as the temperatures continue to increase. The present static seal is the O-ring, which is placed in a standard O-ring groove. This is a very useful object, and an object which the engine designer now uses perhaps too generously. This item will become the victim of further increases in oil system fluid temperatures. Before the designer will be able to part with this device he will have to relearn some of the tricks that were used before O-rings were available. For the present, however, the designer may still benefit from further improvement in materials such as in the new silicone rubber O-rings which are now available. These will allow lubricant temperatures up to a limit of about 450° F.

The present rubber hose on turbojet engines is now being replaced by flexible steel hose in some points, and by a new silicone rubber lined hose elsewhere. The silicone rubber lined hose will handle normal oil system pressures up to 450° F in ambient temperatures of 400° F. Above these temperatures, steel is used.

Fortunately, the newly developed hoses are usually lighter in weight than the old rubber hose that is being discarded, so that the weight penalty of using higher operating temperature fluids is minimized.

A fortunate development has been the fact that the silicone rubber materials are not affected by the diester-base lubricants. However, with the increased interest in silicone lubricating fluids (and the success of some of these in component tests) it becomes evident that the sealing problem with the silicone fluids will be a severe one. Most of the silicone lubricants have an adverse effect on the silicone rubber seals. Steps should be taken now to develop seal materials which will operate to the same limits as the silicone fluids which are compatible with them.

Bearing material development has kept pace reasonably well with the increased operating temperatures up until now. However, the point has been reached where any further increase in lubricating fluid operating temperatures will require thrust bearings to be made from tool steel. There is still a decided lack of knowledge of the fatigue-life limits for these steels. This information is being compiled by the bearing manufacturers, but not at a rapid enough rate.

Gear materials are in a more questionable state than bearing materials. Gears are already being drawn down in hardness in some conditions of operation. This will eventually mean a weight penalty in gear design unless a suitable gear steel is found that will operate with high hardness in the 500° F range. Here again the engine designer will have a choice of whether he should take other steps in the engine design to keep the temperatures of the lubricating fluid down to a reasonable value by one of the means mentioned earlier, whether he should increase weight by operating at a lower hardness with a higher temperature fluid, or whether he should procure a higher-temperature gear steel.

TABLE I. - HEAT LOAD AND HEAT-BALANCE SUMMARY FOR ENGINE OF 10,000 POUNDS THRUST

Altitude, ft	Sea level	Sea level	35,000	35,000	35,000	60,000	60,000
Flight speed							
Mach number	0	1.0	0.53	1.5	1.89	1.0	1.89
Knots	0	660	300	865	1,075	574	1,080
Heat load, Btu/hr							
No. 1 bearing	13,500	13,750	3,310	7,900	8,900	2,675	3,440
No. 1 bearing seal	1,360	1,155	1,650	1,435	1,350	2,010	1,845
No. 2 bearing	41,000	37,500	38,000	38,000	36,500	43,000	36,500
Gearbox	19,100	19,100	19,100	19,100	19,100	19,100	19,100
Lubricant pump	9,880	9,880	9,880	9,880	9,880	9,880	9,880
Variable-displacement pump	9,920	9,920	9,920	9,920	9,920	9,920	9,920
Constant-speed drive	45,500	45,500	45,500	45,500	45,500	45,500	45,500
Total	140,260	136,900	127,360	131,720	131,150	132,085	126,185
Cooling from air flow ^a , Btu/hr	13,000	8,000	19,000	11,500	7,000	17,500	6,500
Engine heat load, Btu/hr	127,260	128,900	108,360	121,220	124,150	114,585	119,685
Temperature of fuel at cooler inlet ^b , °F	126	122	179	130	124	195	159
Temperature of oil at cooler outlet, °F	165	191	322	177	171	362	240

^aCooling due to passage of air over external engine parts.

^bThese temperatures show increase in temperature due to bypass flow through fixed-displacement pumps.

APPENDIX D

TURBOPROP GEAR LUBRICATION PROBLEMS

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The turboprop engine requires: (1) that the lubricant permit starting at -65° F for military applications, (2) that the fluid be resistant to oxidation and other thermal decomposition at bearing operating temperatures to approximately 400° F, and (3) that the lubricant be noncorrosive to metals present in the system. These requirements also apply to the turbojet engine. However, the turboprop engine presents the additional requirement (4) that the lubricant provide adequate load-carrying ability for the reduction-gear system. When it is considered that the gearbox weight may approach one-third of the total weight of the engine, the necessity of designing the gear train to the highest loads permissible in accordance with good practice becomes obvious.

No currently available material has been proven to fulfill all four of these requirements. The present MIL-L-7808 oil does not provide the load-carrying ability desired and may be inadequate in low-temperature properties. A higher-viscosity lubricant (E.E.L. 3A), currently operated in Allison turboprop engines, provides the load-carrying ability and thermal stability required, but is inadequate for -65° F starting. Attack of these lubricants on bearing cages electroplated with lead-base codeposits, has been experienced in engine operation. Laboratory tests, conducted over the 250° to 450° F range, have indicated that this corrosive activity rises sharply with temperature.

The diester-base lubricants also severely affect organic materials with which they are in continuous or incidental contact. Rubber seals and hose, some plastics, and many organic coatings may be seriously attacked by contact with these lubricants. In addition, the higher operating temperatures of engines requiring use of the diester oils render the materials problem more severe. Complete failure of MIL-L-5511 hose, due to degradation and subsequent erosion of the lining, has been experienced in 80 hours with synthetic oil at approximately 250° F.

Other related problems are the scuffing and wear rates of bearing rollers and races at high speeds, and scuffing and pitting fatigue of reduction gearing. The relationship of the lubricant to the scuffing and wear problem on high-speed roller bearings is not known. Mechanical design and metallurgical changes are being explored for solutions to the bearing problem. Previous reduction-gear experience, upon which current designs are based, has been with lubricants of higher viscosity and load-carrying ability. Refinements of the gear system to reduce the lubricant requirements are being made, but for practical application to production, the lubricant should not be marginal in its load-carrying ability. There have been published data to indicate that pitting fatigue life is reduced with lower-viscosity lubricants, but the magnitude as applied to both gears and bearings in practice is not known.

With increasing compression ratios and engine complexity, hot-spot temperatures and, to a degree, bulk operating temperatures of the turboprop engine may be expected to increase. However, the increases are not expected to be as great as for high-speed turbojet aircraft. Increased lubricant load-carrying requirements are indicated if higher ratings resulting from power section development are to be utilized without the accompanying sacrifice in weight or volume requirements for the gear box.

Development of more complex engines such as the two-spool design will result in higher bearing speeds and render the present scuffing problem more severe. Anticipated turboprop bearing temperatures are not expected to render present bearing materials inadequate; hence, developments in this field are primarily directed toward turbojet engines. Some reduction in heat rejected from the gearbox may be experienced with higher-flight-speed turboprop aircraft with supersonic propellers because of reduced reduction-gear-ratio requirements.

The use of more active extreme-pressure-type additives to improve the load-carrying ability of the turboprop lubricant has not been successful to date. The conventional sulfur and chlorine containing additives are corrosive to metals in the high-temperature portions of the power section and cause excessive deposit formations. The use of an acid phosphate improved the load-carrying capacity and provided acceptable cleanliness characteristics. However, the acid phosphate caused the oil to foam excessively and, in addition, chemically attacked copper and also magnesium when moisture was present. It is also noted that the type of antioxidant used in stabilizing the diester lubricants attacks cadmium, preventing the use of this plating material in the oil system.

The present effort, with regard to military applications, is to use one synthetic oil for both turboprop and turbojet engines. This is a worthwhile objective, but the cost in the design compromises required may outweigh the advantages. The present MIL-L-7808 lubricant contains both antiwear agents and a viscosity improver for the benefit of the turboprop. Their presence is undesirable for jet engines as it renders -65° F starting more marginal and prevents use of the engine lubricant as a hydraulic fluid.

It appears that the future lubricant requirements for the turbojet and the turboprop engines may be more difficult to achieve with one oil than at present. The high-temperature requirements of the two engines are currently similar but, principally as a result of supersonic flight speeds, the future turbojet oil temperature levels will be much higher. The necessity of improved load-carrying ability of a given viscosity lubricant must be satisfied for the turboprop engine if low-temperature starting requirements and increased power ratings are to be realized. With present knowledge of lubricant compounding, additives which increase load-carrying ability will limit the high-temperature stability of the lubricant, and the search for better high-temperature lubricants is restricted if improved load-carrying ability, or even that equivalent to the present MIL-L-7808, is a primary requisite. Therefore, separation of the two requirements appears preferable if the desired rate of progress is to be attained.

The use of a separate lubricant for the gearbox system has been considered as a possible solution to the problem. This would be attractive from the viewpoint of lubricant compounding, as conventional extreme-pressure additives might be used. However, inherent disadvantages of this arrangement are serious. Foremost, the addition of the wrong oil to either the gearbox or the power section system could be disastrous. In addition, some existing turboprop engines or airframe installations could not be readily adapted to this change. The requirements for separate oil tanks, additional cooling facilities, and low-temperature starting problems with the use of two lubricating systems render this arrangement undesirable if another solution can be obtained.

The development of two oils, one for satisfying the critical requirements inherent to present and future turbojet engines and the second for the requirements most critical to the turboprop engines, is believed the most desirable approach to the problem and one that offers potential benefits to development of both types of engines. Currently, operation of turboprop engines with a higher-viscosity

lubricant is deemed necessary. However, recent development work on more stable extreme-pressure agents, as conducted by Dr. Fenske of Pennsylvania State College, and lubricants of much improved viscosity-temperature characteristics, such as the silicone-diester blends of the NACA, indicate that material advances in low-temperature properties may be obtainable while maintaining high load-carrying ability. Lubricant improvement by the development of materials with better viscosity-temperature characteristics is considered the more desirable approach, as little experience and information is available concerning wear rates and pitting fatigue which may be experienced with low-viscosity lubricants.

It is emphasized that the lubrication problems of the turboprop engine cannot be solved by lubricant improvement alone. Improvements in both gear and bearing design parameters and materials to provide at least partial solutions are being actively investigated. It appears desirable to modify the lubrication system so as to increase the maximum lubricant viscosity which can be pumped. However, the separation of lubricants for the turbojet and turboprop engines and recognition of adequate load-carrying ability as the primary requisite for the turboprop engine lubricant will materially accelerate development of this power plant.

APPENDIX E

NOTES ON HIGH-TEMPERATURE FLUIDS AND LUBRICANTS

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INTRODUCTION

Development work on high-temperature hydraulic fluids and on high-temperature lubricants for jet aircraft have many things in common. The same requirements of adequate lubricity, fluidity, stability, compatibility, and corrosion resistance are common to both types of fluid. The similarity is such that in practice a single diester type of fluid has been prepared to meet the requirements of MIL-L-6387, the high-temperature hydraulic fluid, and MIL-L-7808, the synthetic jet-engine lubricant. Because of the present state of mechanical developments in hydraulic and lubricating systems, there are somewhat different points and degrees of emphasis on property requirements. The need for an adequate lubricant for high-speed, highly loaded gears in the turboprop engine and in accessory cases of the turbojet engine normally places more emphasis on lubricity of the lubricant than is the case with hydraulic fluids.

Closed hydraulic systems are a current achievement and as such represent a boon to hydraulic-fluid development not currently enjoyed in the jet-engine lubricant field. The use of a closed system in which air may be partially or wholly excluded places the emphasis on thermal stability, or stability after small amounts of oxygen assimilation. These properties are, in general, much less critical than the oxidation stability under conditions where air is freely accessible. These latter conditions appear to dominate in current designs of jet-engine lubrication systems. In general, all the research and development work on high-temperature hydraulic fluids is directly applicable to high-temperature jet-engine lubricants.

HIGH-TEMPERATURE HYDRAULIC FLUIDS

Lubrication and volumetric efficiency are two important items in the operation of hydraulic pumps at high temperatures. Direct measurement of these properties at the desired temperature is very difficult because of the current lack of proven high-temperature pumps. There have been indications that in the temperature range above 300° F mechanical problems with currently available pumps essentially prohibit fluid testing. Another approach to testing lubrication and volumetric efficiency has been used by this laboratory. Lubricity and viscosity of high-temperature hydraulic fluids and lubricants can be measured in laboratory testers. These measured properties at the desired temperatures can be incorporated in an experimental fluid in the temperature range of 100° to 200° F. This experimental fluid can then be tested in a pump under temperature conditions where no mechanical problems are anticipated.

Previous tests conducted by this laboratory have indicated that good quality mineral oil and ester-base hydraulic fluids will operate satisfactorily in piston, gear, and vane-type hydraulic pumps in the temperature range of 100° to 200° F. The minimum viscosity level of these fluids under test conditions was 3.5 centistokes. The current study has been limited, therefore, to viscosity levels below 3.5 centistokes at the test temperature. Preliminary tests have been conducted in a Vickers model PF-17-3911-10ZEL piston pump on this laboratory's number 3 pump test stand.

A comparison of the viscosities and relative volatilities of the mineral oil compositions and typical MIL-L-7808 and MIL-L-6387 type fluids is shown in table I.

The mineral oil fractions PRL 3414 and PRL 3415 exhibit viscosities of 1.0 to 3.3 centistokes in the range of 100° to 200° F. The other test fluids of table I, the ester-base fluids, exhibit this same viscosity range over the temperature range of 250° to 600° F. For esters and mineral-oil fractions of the type shown in table I, the C.O.C. fire point is roughly equivalent to the boiling point of the fluid at a pressure of 10 millimeters of mercury. It is evident, therefore, that the mineral-oil fractions and the ester-base fluids have a similar relation between viscosity level and vapor pressure. That is, tests with the mineral-oil compositions at 100° to 200° F adequately simulate the viscosity level and volatility of typical MIL-L-7808 and MIL-L-6387 fluids at 250° to 600° F. The test fluids PRL 3414 and PRL 3415 of table I are mineral-oil fractions containing an antiwear additive and an oxidation inhibitor. The compositions of these test fluids are shown in table II.

The effect of bulk oil temperature on the wear characteristics of typical mineral oil and ester-type hydraulic fluids and lubricants has been determined with a Shell four-ball wear tester; these wear data, over the temperature range of 165° to 400° F, are shown in table III. These data indicate that the effect of tricresyl phosphate as the antiwear additive persists over the entire temperature range.

There is some indication that the persistence of antiwear properties due to tricresyl phosphate is related to the tricresyl phosphate susceptibility of the base oil. This effect of tricresyl phosphate susceptibility has been discussed in some detail in report PRL 5.4, Sept. 1952. The base stock for PRL 2860 shows better tricresyl phosphate susceptibility than the base stock for PRL 3039. This same trend can be seen in the effectiveness of tricresyl phosphate with temperature rise. It should be emphasized that no substantial change in the effectiveness of tricresyl phosphate would be expected in the temperature range up to about 600° F. In the section of this report dealing with thermal stability, tricresyl phosphate is shown to have good thermal stability up to a temperature of 600° F. These data would indicate that wear properties of mineral oil and ester-base compositions containing tricresyl phosphate are adequate at high temperatures.

Wear characteristics of PRL 3414 and PRL 3415 in the Shell four-ball wear tester are shown in table IV. Wear properties of several ester-base compositions are also shown for comparison. The wear characteristics of the two mineral oil compositions essentially match those of the ester-base fluids containing tricresyl phosphate as the antiwear additive. A good correlation has been shown in the laboratory between wear in gear and vane-type hydraulic pumps and wear at 1- and 10-kilogram loads in the Shell four-ball wear tester. The successful operation of Vickers piston pumps with the same quality lubricants as those evaluated in the gear and vane-type pumps suggests about the same maximum lubricity demands for all three pumps. The wear values for PRL 3414 and PRL 3415 at 1- and 10-kilogram loads and 167° F are about the same as for high-quality mineral-oil and ester-base fluids at 1- and 10-kilogram loads and 400° F. To summarize, it is believed that PRL 3414 and PRL 3415 exhibit the same lubricity, volatility, and viscosity levels between 100° and 200° F as those of MIL-L-7808 and MIL-L-6387 type fluids between 250° and 600° F.

Performance of PRL 3414 and PRL 3415 in the Vickers piston pump is shown in table II. The pump operated satisfactorily throughout this series of tests. It should be emphasized that the pump used in this study is a standard production model. A typical MIL-O-5606 fluid was used as a test fluid to establish a basis for volumetric delivery. The volumetric efficiency as a function of viscosity is shown in table V.

These data indicate that currently available pumps can be operated successfully with relatively low-viscosity, high-volatility fluids provided that good lubricity characteristics are incorporated in the fluid. These data suggest that currently available ester-type fluids are suitable for high-temperature operation in hydraulic pumps provided that current tolerances and lubricity demands can be maintained in the design and construction of high-temperature pumps.

WEAR AND LUBRICATION CHARACTERISTICS OF SILICONES AND SILICATES

Silicones and silicates have been included in the lubrication studies of high-temperature hydraulic fluids. The lubricity properties of mineral-oil and ester-type fluids have already been discussed. Shell four-ball data (from wear and extreme-pressure lubricant tests) for silicone and silicate fluids are compared with similar data for typical ester-type fluids in table VI. The lubricant PRL 3103 is di-2-ethylhexyl sebacate, while PRL 3161 is a complete lubricant formulation of the MIL-L-7808 types. The silicate-base hydraulic fluid, MLO 5277, is a finished composition prepared by the Standard Oil Company of California. The two General Electric silicone fluids are believed to be methylphenyl silicones of good thermal stability.

The finished silicate fluid shows wear characteristics and incipient seizure values of the same order of magnitude as a typical mineral oil or dibasic acid ester without an antiwear additive. Finished fluids of the PRL type are superior to the silicate fluid in this respect. The silicones show very poor steel-on-steel wear and incipient seizure values. The addition of a reasonable quantity (approximately 50 weight percent) of ester to the silicone (PRL 3424) improves the wear and incipient seizure values. In general, a silicone-ester blend of this type exhibits improved response to lubrication additives. PRL 3425 is a similar blend with the same antiwear additives but containing a relatively small amount (approximately 10 weight percent) of the ester. The wear characteristics of PRL 3425 revert to essentially those of the silicone (PRL 3358) itself. It is shown in the section on high-temperature oxidation that the silicone-ester blend PRL 3424 exhibits no better stability than the ester itself. In other words, it appears that in order to improve the wear characteristics of a silicone fluid of the PRL 3358 type by adding an ester, it is necessary to add the ester in sufficient concentration so that the resultant blend assumes the high-temperature oxidation stability of the ester itself.

The effect of temperature on the wear characteristics of silicone and silicate fluids is shown in table VII. Data for a high-quality mineral oil and an ester type fluid are shown for comparison purposes. The wear properties of the silicate fluid PRL 3416 (MLO 5277) are essentially unchanged by temperatures over the range of 165° to 400° F. The silicones show some tendency toward increased wear with increasing temperature.

To summarize, the silicate fluid shows wear and extreme-pressure properties quite similar to those of typical nonadditive mineral-oil and ester fluids. Fluids of this lubricity level have been successfully used as hydraulic fluids. The silicones show very poor lubricity properties.

THERMAL STABILITY OF LUBRICANTS

Thermal-stability tests evaluate the behavior of a fluid or lubricant in the absence of oxygen or under an inert atmosphere such as nitrogen. In systems which are closed, or which provide little contact with air, these tests on thermal

stability are pertinent in showing what happens to a fluid or lubricant. When substantial amounts of oxygen are in contact with the oil, the degradation produced by oxygen usually exceeds that produced by heat or by simple pyrolysis.

Temperature affects thermal stability in a manner similar to that of oxidation, and the temperature coefficient for pyrolytic reactions is similar to that for oxidation reactions. The common metals in bulk form are usually not strong promoters of thermal decomposition.

Table VIII illustrates the effect of 500° F for 20 hours under a nitrogen atmosphere on some esters, ethers, mineral oils, and silicones. As noted, these materials are fairly stable with the exception of esters prepared from secondary-alcohol linkages. The latter decompose much more easily than esters prepared from primary alcohols. Olefins are one of the products of the thermal decomposition of esters. The thermal decomposition of esters made from secondary alcohols becomes significant at 400° to 450° F, and after about 20 hours. On the other hand, esters prepared from primary alcohols will have a comparable stability at 550° to 600° F.

In inspecting all the tables on thermal stability, it is important to remember that decomposition is not always evidenced by an increase in neutralization number. For example, the stability of mineral oils (hydrocarbons), ethers, silicones, and silicates cannot be judged by neutralization number. In these latter cases, changes in viscosity, or the formation of gas or insolubles, are more logical criteria of degradation.

The effects of metals on thermal stability at 500° F for a period of 20 hours is shown in table IX. In general, the metals shown do not greatly accentuate the thermal decomposition. Several of the esters tested show some evidence of steel and magnesium corrosion at 500° F.

Since the 500° F thermal tests did not significantly change the properties of a variety of fluids and lubricants, similar studies were made at 600° F for 6 hours in the absence of metals. These data are presented in table X. Hercoflex 600 which is an ester prepared from pentaerythritol and a monobasic acid, has good stability. Its structure is such that it cannot readily produce an olefin on decomposition. Naphthenic mineral oils, tricresyl phosphate, and some silicates and silicones also are quite stable at 600° F for 6 hours.

Further thermal tests on some of the more promising materials in table X are shown in table XI. These tests were made in a pressure cylinder in order to determine the amount of gaseous or volatile products formed. The polyglycol and the glycol-centered ester show the greatest formation of volatiles and gas. The pressure in the case of 2-ethylhexyl sebacate is due to octene and, as noted, it is condensable to a liquid upon cooling to room temperature. The extent of the decomposition in this pressure test at 600° F is the same as that in the atmospheric tests at 600° F (table X). In this pressure test at 600° F, Hercoflex and tricresyl phosphate together with the silicate and silicone are reasonably stable. Hydrocarbon oils are also quite stable at 600° F.

Table XII shows the thermal stability at 680° to 750° F for 6 hours of several commercially available mineral oils and hydrocarbons. The naphthenic materials at 750° F show sizable losses in viscosity without significant volatility losses. The paraffins are not directly comparable because they are much lower in viscosity. The two materials shown have high pour points. The aromatics, on the other hand, show some viscosity increase at about 700° F.

The effect of temperature on the thermal decomposition of two esters and some naphthenic mineral oils is shown in table XIII. In the case of 2-ethylhexyl sebacate, 6 hours at 600° F produces about 10 percent decomposition to acid and olefin as judged by neutralization number. Accordingly, about 600° F appears to be about the maximum temperature that this ester can tolerate.

As judged by excessive volatile product formation, Hercoflex 600 appears reasonably stable up to 650° to 700° F.

In the case of the naphthenic oils (Nectons), a significant viscosity loss is noted at 700° F and higher. This viscosity loss is not accompanied by any other serious degradation product, such as the formation of volatile products. In a closed system, or where access to air is restricted, some naphthenic-type hydrocarbons appear suitable up to 700° to 750° F. This situation may prevail in a closed high-temperature hydraulic system.

OXIDATION STABILITY OF LUBRICANTS

At temperatures above 450° F, the characteristic induction period of a well inhibited or stabilized oil no longer exists because the oxidation reaction is so rapid. The role of the conventional inhibitor is now that of slowing down oxidation rather than suppressing it entirely for a given interval of time. In studying oxidation at high temperatures, attention is directed, not to an induction period, but to the amount of oxygen assimilated in a given time by a given amount of oil, and to the changes in the properties of the lubricant produced by this amount of oxygen.

Tables XIV to XXIV show the effects of various types of oxidation conditions on certain esters and mineral oils at 500° F.

Table XIV presents data for 2-ethylhexyl sebacate at 500° F and under various conditions of oxidation. Phenothiazine is still active in reducing the over-all effects of oxidation at 500° F. The glass beads serve primarily to increase the contact of air and lubricant.

Table XV shows the behavior of different ester types without catalysts at 500° F, while table XVI shows similar data where glass beads have been used to increase the air-oil contact. This increases the severity of the test. In the case of the phthalate ester, the insolubles are primarily phthalic anhydride and not sludge.

Tables XVII to XIX show oxidation behavior of several lubricants at 500° F with a large area of copper and steel present. One of the striking differences between the effects of glass beads and of copper and steel occurs with the mineral oil designated Voltesso 36. This is a well refined naphthenic oil. Oxidation under these conditions in the presence of the metals resulted in a solid. In general, the esters are affected less by the presence of metals at high temperatures (500° F) than mineral oils.

Table XVIII contains data for three silicate-base fluids and also three silicone oils. The silicates exhibit property changes after oxidation that are comparable to those occurring in the most stable esters. The silicones, however, are very stable.

Table XIX shows oxidation data for a blend of 2-ethylhexyl sebacate and a silicone. This is a high-temperature-type silicone, and such a blend has better lubricating properties than does the silicone alone. However, upon oxidation at 500° F there appears to be little advantage, for the degradative properties of the blend are at least as poor as those of the ester alone.

Tables XX and XXI relate more to hydraulic oils than to high-temperature lubricants for aircraft turbine engines. These tests were made at 500° F with limited air supply (0.6 liter/hr/100 ml of oil). Present as catalysts are 1-inch squares of sheet copper, aluminum, and steel. The reduced air supply is to typify a closed hydraulic system.

Table XX shows oxidation data for some esters, and a silicate and silicone oil. Table XXI contains data for various synthetic hydrocarbons and naphthenic mineral oils. Under these oxidation conditions at 500° F, but with a restricted air supply, most of the fluids have satisfactory properties, including low corrosion. The fluids illustrated represent high-quality products commercially available in each of the classes. It should be noted that further refining with silica gel did not markedly improve the quality.

Phenyl α -naphthylamine (table XXI) as an inhibitor in mineral oils effectively retards the rate of oxidation at 500° F.

Table XXII shows that the properties of an oxidized oil are primarily dictated by the amount of oxygen assimilated by a given amount of oil, and not on the total oxidation time or rate of oxidation. This shows that when oxidation can occur, it usually dominates thermal or pyrolytic effects.

Tables XXIII and XXIV show the effect of oxidizing a mineral oil and an ester to varying degrees at 500° F. The ester can absorb a larger amount of oxygen before its properties resemble those of the oxidized mineral oil.

The preceding discussion on oxidation has been limited to 500° F. The oxidation tests for MIL-L-7808 and MIL-L-6387 fluids are conducted at 347° F. Figure 1 and the following table show the effect of temperature on oxidation (induction period) for a fluid typical of these specifications; the pronounced temperature effect is well illustrated as follows:

Temperature, °F	Stability, hr
347	125
400	27
450	5
500	<1

Petroleum Refining Laboratory
School of Chemistry and Physics
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September 9, 1953

TABLE I. - SOME PROPERTIES OF HIGH-TEMPERATURE HYDRAULIC FLUIDS

Test Fluid	C.O.C. fire point, °F	Viscosity, centistokes			
		3.0 cs	2.0 cs	1.5 cs	1.0 cs
		Temperature at which viscosity is obtained, °F			
PRL 3313	495	400	510	600	---
PRL 3161 (MIL-L-7808 Type)	495	285	360	435	570
PRL 3039 (MIL-L-6387 Type)	425	265	340	415	550
PRL 3414	230	107	145	178	240
PRL 3415	235	72	109	142	203

TABLE II
PUMP PERFORMANCE STUDIES WITH LOW VISCOSITY HYDRAULIC FLUIDS
Tests conducted with Vickers Model PF-17-3911-10ZEL piston pump on PRL #3 pump test stand

Pump Test No.	240	245	246	247	248	249
Test Fluid	PRL 3315	PRL 3414	PRI 3415			
Base Stock, Wt. %	Typical Spec. MIL- O-5606	XCT White Oil ⁽¹⁾ 94.6	Kendall C-13 ⁽²⁾ 94.6			
Antioxidant, Wt. %	Fluid ----	Paranox 441 0.4	Paranox 441 0.4			
Anti-wear Additive, Wt. %	----	Tricresyl Phosphate 5.0	Tricresyl Phosphate 5.0			
Viscosity at 100°F., cs.	14.4	3.27	2.18			
Viscosity at Test Temp., cs.	14.4	3.27	2.18	1.40	1.06	2.18
Pump Speed, r.p.m.	3600	3600	3600	3600	3600	3600
Pump Inlet Temp., °F.	100	100	100	150	200	100
System Pressure, p.s.i.	2900	3000	3000	2950	2750	3000
Test Time, hrs.	52	24	24	11	1	0.5
Average Flow Rate, g.p.m. ⁽³⁾	3.10	3.04	3.00	2.89	2.76	3.00 ⁽⁴⁾

(1) Narrow boiling fraction of highly refined naphthenic gas oil obtained from the Standard Oil Company of New Jersey.

(2) Narrow boiling gas oil from Pennsylvania crude obtained from Kendall Refining Company.

(3) Flow rates are an average for the entire test time.

(4) Tests 246 and 249 were conducted on the same test fluid under the same conditions. The resultant flow rates indicate that the volumetric efficiency has not been altered by tests 247 and 248.

Petroleum Refining Laboratory
School of Chemistry and Physics

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State College, Pennsylvania
January 26, 1953

TABLE III
EFFECT OF BULK OIL TEMPERATURE ON THE WEAR CHARACTERISTICS OF SEVERAL LUBRICANTS
IN THE SHELL FOUR-BALL WEAR TESTER

Test Conditions Include: Test Time = 1 Hour; Test Temperature as Indicated; Test Speed = 850 r.p.m.;
Steel Balls = SKF Industries Grade #1 (0.5-inch Diameter) Steel Ball Bearings, PRL Batch #8.

Test Fluid PRL 3090 = Necton 42, a low pour point mineral oil fraction having 32.3 centistokes
viscosity at 100°F. + 0.4 Wt.% Paranox 441.

PRL 3091 = 1.0 Wt.% Tricresyl Phosphate in PRL 3090.

PRL 2574 = Spec. 51-F-21 type fluid prepared without an anti-wear additive.

PRL 3078 = Spec. 51-F-21 fluid containing 1.0 Wt.% Tricresyl Phosphate.

PRL 2429 = Di-2-Ethylhexyl Sebacate.

PRL 2860 = 1.0 Wt.% Tricresyl Phosphate + 0.4 Wt.% Paranox 441 in Di-2-Ethylhexyl Sebacate.

PRL 3069 = Experimental Gear Box Lubricant prepared by Rohm and Haas. Contains 1.0 Wt.%
Tricresyl Phosphate and 7.5 Wt.% Santopoid S.

PRL 3077 = Experimental ester-base hydraulic fluid prepared without an anti-wear additive.

PRL 3039 = Experimental ester-base hydraulic fluid containing 1.0 Wt.% Tricresyl Phosphate.

PRL No.	- - - - - Average Wear Scar Diameter, mm. for Steel-on-Steel Bearing Surfaces - - - - -														
	1 Kilogram Load					10 Kilogram Load					40 Kilogram Load				
	Test Temperature, °F.					Test Temperature, °F.					Test Temperature, °F.				
	167	266	300	351	399	167	266	300	351	399	167	266	300	351	399
3090	0.22	0.28	0.32	0.38	0.30	0.58	0.58	0.56	0.39	0.35	0.56	0.64	0.84	0.84	-
3091	0.14	0.15	0.17	0.18	0.25	0.33	0.26	0.28	0.28	0.33	0.67	0.84	0.71	0.91	-
2574	0.35	0.44	0.38	0.41	0.33	0.56	0.65	0.56	0.57	0.51	0.66	0.76	0.84	0.93	0.91
3078	0.15	0.16	0.15	0.15	0.14	0.22	0.22	0.25	0.23	0.23	0.44	0.54	0.70	0.97	0.84
2429	0.26	0.49	0.46	0.44	0.52	0.55	0.54	0.58	0.78	0.84	0.60	0.91	0.97	1.04	1.19
2860	0.14	0.17	0.15	0.16	0.18	0.25	0.25	0.26	0.26	0.30	0.38	0.80	0.91	0.97	0.97
3069	0.27	0.32	0.32	0.58	0.57	0.36	0.43	0.47	0.78	0.76	0.50	0.55	0.70	0.91	0.91
3077	0.32	0.46	0.44	0.50	0.58	0.63	0.70	0.81	0.97	1.04	0.84	0.97	0.97	1.10	1.10
3039	0.18	0.16	0.16	0.18	0.20	0.28	0.44	0.59	0.50	0.58	0.91	0.91	0.97	0.84	0.74

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School of Chemistry and Physics

The Pennsylvania State College
State College, Pennsylvania
January 26, 1953

TABLE IV
WEAR CHARACTERISTICS OF SOME LOW VISCOSITY HYDRAULIC FLUIDS

Tests Conducted in the Shell Four-Ball Wear Tester

Test Conditions Include: Test Time = 1 hr.; Test Temp. = 75°C.;

Test Speed = 850 r.p.m.; Steel Balls = SKF Industries Grade #1

(0.5 inch Diameter) Steel Ball Bearings, PRL Batch #10

PRL No.	Test Fluid Composition in Wt. %	Visc. at 100°F., cs.	Average Wear Scar Diameter, mm. Steel-on-Steel Bearing Surfaces		
			1 Kg.	10 Kg.	40 Kg.
2429	Di-2-ethylhexyl Sebacate	12.6	0.26	0.55	0.60
2860	0.4 Paranox 441 + 1.0 Tricresyl Phosphate in PRL 2429	12.7	0.14	0.25	0.38
3161	3.9 Acryloid HF-25 + 0.5 Phenothiazine + 5.0 Tricresyl Phosphate in PRL 2429	22.5	0.13	0.22	0.38
3414	0.4 Paranox 441 + 5.0 Tricresyl Phosphate in XCT White Oil ⁽¹⁾	3.27	0.14	0.20	0.44
3415	0.4 Paranox 441 + 5.0 Tricresyl Phosphate in Kendall C-13 ⁽²⁾	2.18	0.15	0.48	0.45

(1) Narrow boiling fraction of highly refined naphthenic gas oil obtained from the Standard Oil Company of New Jersey.

(2) Narrow boiling fraction of Pennsylvania gas oil obtained from the Kendall Refining Company.

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The Pennsylvania State College
State College, Pennsylvania
January 26, 1953

TABLE V. - VOLUMETRIC EFFICIENCY VERSUS
FLUID VISCOSITY IN A VICKERS
PF-17-3911-10ZEL PISTON PUMP

Fluid viscosity. centistokes	Volumetric delivery, gal/min	Percent of maximum ^a delivery
14.2	3.10	100
3.3	3.04	98
2.2	3.00	97
1.4	2.89	93
1.1	2.76	89

^aVolumetric delivery with typical
MIL-O-5606 fluid at 100° F is consid-
ered 100 percent of maximum delivery.

TABLE VI
WEAR CHARACTERISTICS OF SEVERAL FLUIDS

TEST CONDITIONS INCLUDE: FOUR-BALL WEAR TESTER: TEST TIME = 1 HOUR; TEST TEMPERATURE = 75°C.; TEST SPEED = 850 R.P.M.
EXTREME-PRESSURE LUBRICANT TESTER: TEST TIME = 1 MINUTE; TEST TEMPERATURE = ROOM TEMPERATURE (70-80°F.); TEST SPEED = 1750 R.P.M.
STEEL BALLS = SKF INDUSTRIES GRADE #1 (0.5 INCH DIAMETER) STEEL BALL BEARINGS, PRL BATCH #10.

		- - - - WEAR TESTER - - - -			EXTREME-PRESSURE LUBRICANT TESTER - - - - -										
PRL NO.	TEST FLUID COMPOSITIONS IN WT. %	AVERAGE WEAR SCAR DIAMETER, MM. STEEL-ON-STEEL BEARING SURFACES			APPROX. LOAD FOR INCIPIENT SEIZURE, KG.	AVERAGE WEAR SCAR DIAMETER, MM. STEEL-ON-STEEL BEARING SURFACES									
		1 KG.	10 KG.	40 KG.		40 KG.	50 KG.	60 KG.	70 KG.	80 KG.	100 KG.	120 KG.	140 KG.	160 KG.	
3103	DI-2-ETHYLHEXYL SEBACATE	0.34	0.56	0.69	50	0.39	-	2.07	-	4.55	WELDED				
3161	SPEC. MIL-L-7808 TYPE FLUID	0.16	0.22	0.37	80	0.34	-	0.36	-	2.66	2.88	WELDED			
3382	SILICATE-BASE HYDRAULIC FLUID (MLO 5277)	0.38	0.61	0.71	50	0.36	-	2.01	-	2.73	3.05	2.99	3.25	WELDED	
3416	SILICATE-BASE HYDRAULIC FLUID (MLO 5277)	-	-	-	50	0.36	0.69	1.90	-	-	-	2.99	WELDED		
3357	G.E. SILICONE FLUID, SAMPLE NO. 370-30-183	0.33	2.27	3.77	<40	2.14	2.49	3.16	-	2.79	WELDED				
3358	G.E. SILICONE FLUID, SAMPLE NO. 370-30-184	0.33	1.36	2.60	40	0.78	2.49	2.99	-	2.99	WELDED				
1836	A METHYL SILICONE	0.32	0.39	1.40	<40	1.36	-	-	-	2.34	-	WELDED			
-	1.0 TRICRESYL PHOSPHATE IN PRL 1836	0.35	0.47	1.69	-	-	-	-	-	-	-	-			
-	0.01 PHOSPHORIC ACID IN PRL 1836	0.38	0.44	1.77	-	-	-	-	-	-	-	-			
3424	0.5 PHENOTHIAZINE + 1.0 TRICRESYL PHOSPHATE IN 50:50 MIXTURE OF PRL 3358 AND DI-2-ETHYLHEXYL SEBACATE	0.26	0.40	0.61	70	0.38	-	0.42	1.77	3.08	WELDED				
-	0.5 DIMETHYL HYDROGEN PHOSPHITE IN PRL 3424	0.15	0.39	0.58	80	0.42	-	0.49	0.56	1.99	2.73	2.97	WELDED		
3425	0.5 PHENOTHIAZINE + 1.0 TRICRESYL PHOSPHATE + 0.5 DIMETHYL HYDROGEN PHOSPHITE + 11.4 DI-2-ETHYLHEXYL SEBACATE IN PRL 3358	0.15	1.38	2.34	40	1.06	-	2.94	-	2.60	2.51	WELDED			

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THE PENNSYLVANIA STATE COLLEGE
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FEBRUARY 4, 1953

TABLE VII
EFFECT OF BULK OIL TEMPERATURE ON THE WEAR CHARACTERISTICS OF SEVERAL FLUIDS
IN THE SHELL FOUR-BALL WEAR TESTER

Test Conditions Include: Test Time = 1 Hour; Test Temperature as Indicated; Test Speed = 850 r.p.m.;
Steel Balls = SKF Industries Grade #1 (0.5 inch Diameter) Steel Ball Bearings, PRL Batch #10.
Test Fluid: PRL 3078 = Spec. 51-F-21 Fluid (Acryloid-Mineral Oil Blend Containing 1.0 Wt.% Tricresyl Phosphate).
PRL 2860 = 1.0 Wt.% Tricresyl Phosphate + 0.4 Wt.% Paranox 441 in Di-2-Ethylhexyl Sebacate.
PRL 3416 = Silicate Base Hydraulic Fluid Received from the Wright Air Development Center (WADC Designation MLO 5277).
PRL 3357 = General Electric Silicone Fluid, Sample Number 370-30-183.
PRL 3358 = General Electric Silicone Fluid, Sample Number 370-30-184.

PRL No.	- - - - - Average Wear Scar Diameter, mm. for Steel-on-Steel Bearing Surfaces - - - - -														
	1 Kilogram Load					10 Kilogram Load					40 Kilogram Load				
	Test Temperature, °F.					Test Temperature, °F.					Test Temperature, °F.				
	167	266	300	351	399	167	266	300	351	399	167	266	300	351	399
3078	0.15	0.16	0.15	0.15	0.14	0.22	0.22	0.25	0.23	0.23	0.44	0.54	0.70	0.97	0.84
2860	0.14	0.17	0.15	0.16	0.18	0.25	0.25	0.26	0.26	0.30	0.38	0.80	0.91	0.97	0.97
3416	0.37	0.24	0.34	0.34	0.34	0.61	0.61	0.67	0.58	0.53	0.65	1.17	1.04	1.17	1.04
3357	0.33	0.62	0.78	1.04	1.04	2.27	2.53	2.55	2.73	2.60	3.77	3.92	3.79	4.18	-
3358	0.33	0.44	0.43	0.51	0.61	1.36	1.45	1.32	1.52	1.36	2.60	2.70	2.70	2.60	2.47

Petroleum Refining Laboratory
School of Chemistry and Physics
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State College, Pennsylvania
January 29, 1953

TABLE VIII
THERMAL STABILITY OF VARIOUS FLUIDS AT 500°F.

TESTS ARE CONDUCTED AT $500 \pm 5.0^\circ\text{F}$. FOR 20 HOURS USING A TWENTY-FIVE GRAM CHARGE OF TEST FLUID. THE TEST TUBES USED ARE SEALED WITH A U TUBE CONTAINING APPROXIMATELY 3 ML. OF THE TEST FLUID. THE SEAL ALLOWS PASSAGE OF GAS OUT OR AIR IN WITH PRESSURE CHANGES GREATER THAN 4 INCHES OF FLUID-- APPROXIMATELY 1/4 LB./SQ.IN. PRIOR TO THE START OF THE TEST, THE VOLUME OF THE SEALED TUBE IS REPLACED APPROXIMATELY 4 TIMES WITH NITROGEN. THE NITROGEN IS INTRODUCED ABOVE THE SURFACE OF THE TEST FLUID BY MEANS OF A SEALED-IN CAPILLARY TUBE.

PRL NO.	FLUID	CENTISTOKE VISCOSITY 100°F.			% LOSS IN WT. (1)	NEUTRALIZATION NUMBER			% DECOMPOSITION BASED ON THEORETICAL NEUT. NO. OF DIBASIC ACID	ASTM COLOR	
		ORIG.	FINAL	CHANGE		ORIG.	FINAL	MANOMETER FLUID		ORIG.	FINAL
3103	DI-2-ETHYLHEXYL SEBACATE	12.8	12.8	0.0	2.0	0.1	2.8(2)	0.1	1.1	2-1/2	2-1/2
2813	DI-2-ETHYLHEXYL AZELATE	10.8	11.1	+3.0	0.0	0.3	3.0	0.6	1.1	1-1/2	4
2800	DI-2-ETHYLHEXYL ADIPATE	8.11	8.22	+1.3	2.0	0.0	3.2	0.2	1.1	1	2-1/2
2956	"ISOOCTYL" ADIPATE (PLEXOL 244)	9.99	10.0	+0.1	0.0	0.2	5.0(3)	0.2	1.6	2	2-1/2
2996A	DI-3-METHYL BUTYL ADIPATE	4.87	5.06	+3.9	4.0	0.2	7.9	0.4	2.0	1	1-1/2
2975	DI-2-ETHYLHEXYL PHTHALATE	29.7	26.1	-12.1	6.0	0.1	2.8(2)	0.1	1.0	1	2-1/2
3265	DI CAPRYL SEBACATE	13.2	7.57	-42.5	4.0	0.1	80.1(2)	-	30.5	1	(5)
3017	DI-SEC-AMYL SEBACATE	7.52	-	-	8.0	0.0	156.0	0.1	43.9	1	(5)
2850	DI-BUTOXYETHYL SEBACATE	10.8	8.81	-18.1	2.0	1.6	15.5	1.3	5.6	2-1/2	3-1/2
3057	HERCOFLEX 600	22.1	22.4	+1.3	2.0	0.0	0.4	0.1	0.1	1-1/2	2-1/2
3176	TRIETHYLENE GLYCOL DI-2-ETHYLHEXANOATE	8.61	8.57	-0.5	2.0	0.2	0.9	0.3	0.3	1	4
3177	POLYETHYLENE GLYCOL DI-2-ETHYLHEXANOATE	11.0	11.2	+2.0	4.0	0.8	1.3	1.1	0.4	2	7
3311	POLYGLYCOL 18*	17.6	14.2	-19.0	2.0	0.1	9.1	-	-	1-1/2	2-1/2
3300	POLYGLYCOL E-300*	35.8	36.2	+1.8	0.0	-0.4	-0.1(4)	-	-	1	4
3301	POLYGLYCOL P-400*	33.3	32.6	-2.0	2.0	0.1	0.0	-	-	1	5
2973	VOLTESO 36*	9.50	9.59	+1.0	0.0	0.1	0.2	0.1	-	1/2	1/2
2031	NECTON 37*	15.5	15.8	+1.8	2.0	0.3	0.3	0.2	-	2.0	2-1/2
2092	SILICONE NO. 500*	43.4	44.9	+3.5	2.0	0.0	0.0	-	-	1	1
3325	SILICONE NO. 701*	7.28	7.45	+2.3	0.0	0.0	0.0	-	-	1	1
-	TRICRESYL PHOSPHATE*	38.3	41.4	+8.1	2.0	0.2	3.5	0.2	-	2	4

* THESE FLUIDS DO NOT CONTAIN PHENOTHIAZINE. THE REMAINDER OF THE FLUIDS CONTAIN 0.5 WEIGHT PER CENT PHENOTHIAZINE.

- (1) PER CENT LOSS IN WT. IS GOOD TO $\pm 2.0\%$ OR 0.5 GRAMS.
- (2) CRYSTALS FORMED AROUND THE TOP OF THE TUBE WHICH WAS EXPOSED TO ROOM TEMPERATURE. CRYSTALS IDENTIFIED AS SEBACIC ACID IN PRL 3103 AND PRL 3265, AND PHTHALIC ANHYDRIDE IN PRL 2975.
- (3) SMALL AMOUNT OF UNIDENTIFIABLE SLUDGE PRESENT IN THE LIQUID.
- (4) ORIGINAL FLUID AND FLUID FOLLOWING TEST ARE BASIC.
- (5) SOLID PORTION IDENTIFIED AS SEBACIC ACID.

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MARCH 8, 1952

TABLE IX
THERMAL STABILITY TESTS AT 500°F. WITH METAL CATALYSTS PRESENT

TESTS ARE CONDUCTED AT $500 \pm 5.0^\circ\text{F}$. FOR 20 HOURS USING A FIFTY GRAM CHARGE OF TEST FLUID. THE TEST TUBES USED ARE SEALED WITH A U TUBE CONTAINING APPROXIMATELY 3 ML. OF THE TEST FLUID. THE SEAL ALLOWS PASSAGE OF GAS OUT OR AIR IN WITH PRESSURE CHANGES GREATER THAN 4 INCHES OF FLUID - APPROXIMATELY 1/4 LB./SQ. IN. PRIOR TO THE START OF THE TEST, THE VOLUME OF THE SEALED TUBE IS REPLACED APPROXIMATELY 4 TIMES WITH NITROGEN. THE NITROGEN IS INTRODUCED ABOVE THE SURFACE OF THE TEST FLUID BY MEANS OF A SEALED-IN CAPILLARY TUBE. CATALYST = A 1 INCH SQUARE EACH OF THE INDICATED METALS.
NOTE: ALL FLUIDS EXCEPT THOSE INDICATED BY AN ASTERISK (*) CONTAIN 0.5 WEIGHT PER CENT PHENOTHIAZINE.

PRL NO.	FLUID	CENTISTOKE VISCOSITY 100°F.			% LOSS IN WT. (1)	CHANGE IN CATALYST WT., MG./SQ. CM.					NEUTRALIZATION NUMBER, MG. KOH/GM. OIL			WT. % SLUDGE
		ORIG.	FINAL	% CHANGE		CU	STEEL	AL	MG	AG	ORIG.	FINAL	MANOMETER	
3103	DI-2-ETHYLHEXYL SEBACATE	12.9	12.5	-3.1	0.0	-0.01	-0.49	0.00	-0.80	-0.02	0.1	2.8	0.1	0.1
3045	DI-2-ETHYLHEXYL SEBACATE (DISTILLED GRADE)	12.3	12.5	+1.6	1.0	0.00	+1.14	+0.05	-1.39	0.00	0.0	1.6	0.1	TRACE
3107	DI-2-ETHYLHEXYL PHTHALATE	30.1	24.6	-18.3	0.0	+0.11	+0.06	0.00	0.00	0.00	0.0	5.0	0.0	NONE
2813	DI-2-ETHYLHEXYL AZELATE	10.8	10.6	-1.9	1.0	+0.08	-0.12	0.00	-2.52	0.00	0.3	0.6	0.8	TRACE
3057	HERCOFLEX 600	22.1	21.9	-0.9	1.0	0.00	-0.80	0.00	-8.75	-0.01	0.0	5.8	0.1	0.3
3176	TRIETHYLENE GLYCOL DI-2-ETHYL- HEXOATE	8.61	8.73	+1.4	1.0	-0.08	-0.08	-0.03	-0.48	-0.03	0.2	1.3	0.1	1.8
2996-A	DI-3-METHYLBUTYL ADIPATE	4.87	4.68	-3.9	5.4	0.00	-0.28	0.00	-1.75	0.00	0.2	2.0	0.3	NONE
2973	VOLTESSO-36*	9.49	9.58	+0.9	0.0	+0.10	0.00	-0.01	+0.02	-0.03	0.1	0.2	0.1	NONE
3325	SILICONE 701 FLUID (DOW CORNING)	7.28	7.45	+2.3	0.0	+0.02	0.00	+0.01	-	-	0.0	0.0	-	NONE
3161	BATCH 5380 (SPEC. MIL-L-7808 TYPE GEAR LUBE)	22.3	18.0	-19.3	0.0	0.00	+0.02	0.00	-	-	0.2	3.2	0.2	-
	TRICRESYL PHOSPHATE*	38.3	41.0	+7.1	0.0	+0.01	0.00	0.00	-	-	0.2	6.3	0.1	-

(1) PER CENT LOSS IN WEIGHT IS GOOD TO ± 1.0 PER CENT OR 0.5 GRAMS.

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JANUARY 28, 1953

TABLE X

THERMAL STABILITY OF VARIOUS FLUIDS AT 600°F.

TESTS ARE CONDUCTED AT $600 \pm 5^\circ\text{F}$. FOR 6 HOURS USING A TWENTY-FIVE GRAM CHARGE OF TEST FLUID. THE TEST TUBES USED ARE SEALED WITH A U TUBE CONTAINING APPROXIMATELY 3 ML. OF THE TEST FLUID. THE SEAL ALLOWS PASSAGE OF GAS OUT OR AIR IN WITH PRESSURE CHANGES GREATER THAN 4 INCHES OF FLUID - APPROXIMATELY 1/4 LB./SQ. IN. PRIOR TO THE START OF THE TEST, THE VOLUME OF THE SEALED TUBE IS REPLACED APPROXIMATELY 4 TIMES WITH NITROGEN. THE NITROGEN IS INTRODUCED ABOVE THE SURFACE OF THE TEST FLUID BY MEANS OF A SEALED-IN CAPILLARY TUBE.

PRL NO.	FLUID	CENTISTOKE VISCOSITY AT 100°F.			% LOSS IN WEIGHT	NEUT. NO. (MG. KOH/GM. OIL)	
		ORIGINAL	FINAL	% CHANGE		ORIG.	FINAL
3371	DI-2-ETHYLHEXYL SEBACATE	12.7	11.3	-12.8	2.0	0.1	25.3
2813	DI-2-ETHYLHEXYL AZELATE	10.8	9.48	-12.3	0.0	0.3	31.0
2800	DI-2-ETHYLHEXYL ADIPATE	8.11	- (1)	-	0.0	0.0	28.0
2956	"ISOOCYL" ADIPATE (PLEXOL 244)	9.99	7.95	-20.4	10.0	0.2	50.0
2996A	DI-3-METHYLBUTYL ADIPATE	4.87	5.84	+19.8	9.0	0.2	45.0
2975	DI-2-ETHYLHEXYL PHTHALATE	29.7	SOLID	-	20.0	0.3	77.1
-	DI-2-ETHYLHEXYL TEREPHTHALATE	33.9	SOLID	-	20.0	0.1	135.0
2850	DI-BUTOXYETHYL SEBACATE	10.8	8.54	-21.1	4.0	1.6	27.3
3057	HERCOFLEX 600	22.1	22.4	+1.4	0.0	0.1	2.1
3176	TRIETHYLENE GLYCOL DI-2-ETHYL- HEXANOATE	8.61	7.22	-16.3	4.0	0.8	8.4
3177	POLYETHYLENE GLYCOL DI-2-ETHYL- HEXANOATE	11.0	8.7	-20.5	9.0	0.2	6.4
3300	POLYGLYCOL E-300	35.8	35.3	-1.4	2.0	0.0	0.2
3301	POLYGLYCOL P-400	33.3	27.8	-16.5	8.0	0.1	0.6
2803	UCON LUBRICANT 50-HB-55	9.04	12.2	+34.6	25.0	0.2	0.2
2978	UCON LUBRICANT DLB-68-ER	11.0	8.5	-22.8	39.0	8.6	1.6
2801	UCON LUBRICANT LB-135	28.2	15.0	-46.7	31.0	0.6	3.5
2802	UCON LUBRICANT LB-1145	238.1	32.4	-86.0	30.0	0.4	4.0
-	NECTON 42	32.4	32.9	+1.3	2.0	0.2	0.2
-	O-TERPHENYL	4.53 (2)	4.49	-0.9	2.0	0.1	0.1
-	TRICRESYL PHOSPHATE	38.4	41.8	+8.4	0.0	0.2	2.8
3416	SILICATE FLUID (MLO 5277)	27.9	24.3	-12.9	2.0	0.1	0.0
3325	SILICONE FLUID (DOW CORNING)	7.28	7.80	+7.2	0.0	0.1	0.3

(1) INSUFFICIENT FLUID REMAINING FOR VISCOSITY DETERMINATION.

(2) VISCOSITIES DETERMINED AT 210°F.

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FEBRUARY 24, 1953

TABLE XI

THERMAL STABILITY OF VARIOUS FLUIDS IN A STAINLESS STEEL CYLINDER AT 600°F.

TESTS CONDUCTED AT $600 \pm 5^\circ\text{F}$. FOR A PERIOD OF 6 HOURS USING A FLUID CHARGE OF 20 ML. THE TOTAL VOLUME OF THE STAINLESS STEEL CYLINDER IS 46 ML. PRIOR TO STARTING THE TEST THE SYSTEM IS PURGED WITH NITROGEN FOR A PERIOD OF 15 MINUTES. THE SYSTEM IS THEN SEALED AND THE TEST STARTED. THE PRESSURE OF THE NITROGEN AT 600°F. IS ABOUT 20 P.S.I. GAGE.

PRL NO.	FLUID	CENTISTOKE VISCOSITY AT 100°F.			NEUT. NO. (MG. KOH/GM. OIL)		MAXIMUM SYSTEM PRESSURE P.S.I.	FINAL PRESSURE AT ROOM TEMPERATURE
		ORIG.	FINAL	% CHANGE	ORIG.	FINAL		
3371	D1-2-ETHYLHEXYL SEBACATE	12.7	11.9	-6.3	0.1	31.5	72	0
3176	TRIETHYLENE GLYCOL D1-2- ETHYLHEXANOATE	8.61	7.41	-13.8	0.8	8.3	144	33
3057	HERCOFLEX 600	22.1	21.6	-2.3	0.1	7.2	35	6
3300	POLYGLYCOL E-300	35.8	30.5	-14.8	-0.4	0.7	247	49
-	TRICRESYL PHOSPHATE	38.4	51.1	+33.0	0.2	12.9	30	0
3416	SILICATE FLUID (MLO 5277)	27.9	16.6	-40.5	0.1	0.1	35	0
3325	SILICONE FLUID (DOW CORNING)	7.28	7.72	+6.0	0.1	0.0	36	6

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MARCH 25, 1953

TABLE XII
THERMAL STABILITY OF VARIOUS TYPES OF HYDROCARBONS AT HIGH TEMPERATURES

TESTS ARE CONDUCTED FOR A 6 HOUR PERIOD USING A 25 GRAM CHARGE OF TEST FLUID. THE TEST TUBES ARE SEALED WITH A U-TUBE CONTAINING APPROXIMATELY 3 ML. OF TEST FLUID. THE SEAL ALLOWS PASSAGE OF GAS OUT OR AIR IN WITH PRESSURE CHANGES GREATER THAN 4 INCHES OF FLUID - APPROXIMATELY 1/4 LB./SQ. IN. PRIOR TO THE START OF THE TEST THE VOLUME OF THE SEALED TUBE IS REPLACED APPROXIMATELY 4 TIMES WITH NITROGEN. THE NITROGEN IS INTRODUCED ABOVE THE SURFACE OF THE TEST FLUID BY MEANS OF A SEALED-IN CAPILLARY TUBE.

TEST FLUID	TEST TEMP., °F.	CENTISTOKE VISCOSITY AT 100°F.			% LOSS IN WEIGHT	NEUT. NUMBER (MG. KOH/GM. OIL)	
		ORIG.	FINAL	% CHANGE		ORIG.	FINAL
NAPHTHENIC MINERAL OILS							
NECTON 42 (ESSO)	750-700(1)	32.3	16.2	-50	2	0.0	0.1
NECTON 45 (ESSO)	740-690(1)	44.7	18.0	-60	4	0.1	0.1
NECTON 55 (ESSO)	750-710(1)	98.0	31.0	-68	0	0.0	0.0
HUMBLE PALE D (ESSO)	740-690(1)	201	42.7	-79	4	0.1	0.4
PARAFFINIC MINERAL OILS							
EICOSANE (ATLANTIC REFINING)	680 ± 20	2.01(2)	2.00(2)	-1	0	0.0	0.0
WHITE SCALE WAX (KENDALL REFINING CO.)	750 ± 20	3.42(2)	3.28(2)	-4	13	0.0	0.1
AROMATIC MINERAL OILS							
POLYAMYL NAPHTHALENES	700 ± 20	139	135	+1	0	0.0	0.4
SOVOLOID (SOCONY-VACUUM)	700 ± 20	44.9	53.0	+18	4	0.0	0.2
OLA TAR (ESSO)	700 ± 20	48.1	82.4	+71	2	0.1	0.1

(1) TEST TEMPERATURE IS DETERMINED BY THE TEMPERATURE AT WHICH REFLUX IS NOTED WITH THE TEST FLUID. AS DECOMPOSITION OCCURS DURING THE TEST, THE TEST TEMPERATURE IS REDUCED SO THAT A GENTLE REFLUX IS MAINTAINED IN THE PART OF THE TEST TUBE WHICH IS NOT IMMERSSED IN THE HIGH TEMPERATURE BATH.

(2) CENTISTOKE VISCOSITY OBTAINED AT 210°F.

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MAY 15, 1953

TABLE XIII

THERMAL STABILITY OF SEVERAL FLUIDS

TESTS ARE CONDUCTED FOR A PERIOD OF 6 HOURS USING A 25 GRAM CHARGE OF TEST FLUID. THE TEST TUBES ARE SEALED WITH A U-TUBE CONTAINING APPROXIMATELY 3 ML. OF TEST FLUID. THE SEAL ALLOWS PASSAGE OF GAS OUT OR AIR IN WITH PRESSURE CHANGES GREATER THAN 4 INCHES OF FLUID - APPROXIMATELY 1/4 LB./SQ. INCH. PRIOR TO THE START OF THE TEST, THE VOLUME OF THE SEALED TUBE IS REPLACED APPROXIMATELY 4 TIMES WITH NITROGEN. THE NITROGEN IS INTRODUCED ABOVE THE SURFACE OF THE TEST FLUID BY MEANS OF A SEALED-IN CAPILLARY TUBE.

PRL NO.	FLUID	TEST TEMP., °F.	CENTISTOKE VISCOSITY			% LOSS IN WEIGHT	NEUT. NUMBER (MG. KOH/GM. OIL)	
			ORIG.	AT 100°F. FINAL	% CHANGE		ORIG.	FINAL
3103	DI-2-ETHYLHEXYL SEBACATE	500	12.8	12.8	0	2	0.1	2.8
3371	DI-2-ETHYLHEXYL SEBACATE	600	12.7	11.3	-13	2	0.1	25.3
3371	DI-2-ETHYLHEXYL SEBACATE	650	12.7	SOLID	-	5	0.1	126
3057	HERCOFLEX 600	500	22.1	22.4	+1	2	0.1	0.4
3057	HERCOFLEX 600	600	22.1	22.4	+1	0	0.1	2.1
3057	HERCOFLEX 600	650	22.1	23.1	+5	0	0.1	14.2
3057	HERCOFLEX 600	700	22.1	27.1	+23	16(1)	0.1	6.8
2031	NECTON 37	500	15.5	15.8	+2	2	0.3	0.3
2053	NECTON 42	600	32.4	32.9	+1	2	0.2	0.2
2054	NECTON 55	650	98.0	85.9	-12	0	0.0	0.2
2053	NECTON 42	700	32.4	16.2	-50	2	0.2	0.2
2054	NECTON 55	750	98.0	31.0	-68	0	0.0	0.4

(1) APPROXIMATELY HALF OF THIS LOSS FROM THE THERMAL STABILITY TUBE WAS RECOVERED IN A DRY ICE TRAP.

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JUNE 30, 1953

TABLE XIV
OXIDATION BEHAVIOR OF DI-2-ETHYLHEXYL SEBACATE AT 500°F.

PROCEDURES AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-6987

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = $500 \pm 5^\circ\text{F}$; TEST TIME = 6 HOURS; AMOUNT OF TEST FLUID AS INDICATED; AIR RATE AS INDICATED; CATALYST = GLASS WALLS OF TEST CONTAINER ONLY EXCEPT FOR CASES NOTED. IN CASES HAVING AN ADDITIONAL CATALYST, NOT ONLY IS THE CATALYST AREA INCREASED BUT THE AIR TENDS TO BE BETTER DISPERSED RESULTING IN MORE INTIMATE CONTACT BETWEEN AIR AND FLUID.

TEST FLUID = DI-2-ETHYLHEXYL SEBACATE WITH OR WITHOUT PHENOTHIAZINE AS NOTED.

AIR RATE, LITERS PER HOUR	5	5	10	10	10	10	10	10	28	10	10	10	10
PHENOTHIAZINE, WT. %	0.5	NONE	0.5	NONE	NONE	NONE	NONE	NONE	0.5	NONE	0.5	NONE	0.5
ADDITIONAL CATALYST	NONE	NONE	NONE	NONE	NONE	NONE	NONE	NONE	NONE	NONE	NONE	NONE	NONE
TYPE	-	-	-	-	-	-	-	-	(3)	-	-	-	-
AREA, SQ. CM.	-	-	-	-	-	-	-	-	-	-	-	-	-
							GLASS BEADS	GLASS BEADS		STEEL CHAIN	STEEL CHAIN	CU CHAIN	CU CHAIN
							855	755		2000	2000	1185	1410
LIQUID CHARGED, GRAMS	88.0	88.0	89.0	89.5	90.0	90.0	46.0	70	135	94	94	89	99
LIQUID IN TUBE FOLLOWING TEST, GRAMS	87.5	87.0	87.0	82.5	82.0	82.5	37.0	66	-	-	82	79	87
APPROX. AMOUNT OF OXYGEN SUPPLIED, GRAMS ⁽¹⁾	7.8	7.8	15.7	15.7	15.7	15.7	15.7	15.7	46.5	15.7	15.7	15.7	15.7
APPROX. AMOUNT OF OXYGEN USED, GRAMS ⁽¹⁾	0.9	1.9	2.6	4.6	-	4.2	8.0	5.3	15.7	10.0	10.7	8.4	8.4
APPROX. MOLES OF OXYGEN USED/MOLE OF ESTER	0.14	0.29	0.38	0.66	-	0.62	2.3	1.06	1.53	1.41	1.50	1.24	1.13
VOLATILE PRODUCTS IDENTIFIED AS ⁽²⁾													
CARBON DIOXIDE, GRAMS	0.3	1.8	0.6	2.2	2.2	2.0	3.0	1.3	9.6	3.9	3.3	3.5	4.0
WATER, GRAMS	0.9	1.3	1.4	2.2	1.9	1.9	6.2	2.3	18.8	6.1	5.3	4.7	4.5
HYDROCARBON, GRAMS	0.6	3.2	0.8	4.8	3.4	3.9	3.7	1.8	17.8	7.5	6.5	9.1	4.9
TOTAL VOLATILE PRODUCTS, GRAMS	1.8	6.3	2.8	9.2	7.5	7.8	12.9	5.4	46.2	17.5	15.1	17.3	13.4
PROPERTIES OF LIQUID PRODUCT FROM TUBE:													
CHANGE IN VISCOSITY AT													
130°F.	+8	+8	+13	+17	+21	+20	+184	+44	SOLID	+404	SOLID	+102	+60
0°F.	+15	+21	+26	+42	+52	+46	+618	+115	SOLID	+1000	SOLID	+195	+105
NEUT. NO. (MG. KOH/GM. OIL):													
ORIGINAL	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
FINAL	5.5	15.7	8.6	19.0	19.6	14.5	34	28	28	18	27	8	9
ASTM UNION COLOR:													
ORIGINAL	2	2	2	2	2	2	2	2	2	2	2	2	2
FINAL	>8	>8	>8	>8	>8	>8	>8	>8	>8	>8	>8	>8	>8
WT. % INSOLUBLE MATERIAL	TRACE	TRACE	TRACE	TRACE	TRACE	0.02	TRACE	6.5	-	3.5	4.4	1.7	4.5
PROPERTIES OF VOLATILE PRODUCTS:													
WATER LAYER:													
NEUT. NO.	-	99	50	-	-	107	162	52	97	49	33	44	37
HYDROCARBON LAYER:													
NEUT. NO.	-	10	12	-	26	20	39	18	63	19	20	32	22
BROMINE NO.	-	111	74	-	85	84	56	42	9	36	21	29	20

- (1) AMOUNT OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HR.) X O₂ CONTENT (FRACTION) X 1.43 (GM./LITER). AMOUNT OF OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O₂. THE AMOUNT OF OXYGEN USED IS PLOTTED AGAINST TIME. (SEE FIGURES 4, 5, AND 6)
- (2) VOLATILE PRODUCTS ARE COLLECTED BY PASSING EXHAUST GASES THROUGH A DRY ICE TRAP, DRIERITE, ASCARITE, AND ACTIVATED CHARCOAL. IT HAS BEEN DETERMINED THAT THE AIR USED DOES NOT CONTAIN ANY APPRECIABLE AMOUNTS OF WATER OR CARBON DIOXIDE.
- (3) TEST CARRIED OUT IN HIGH-PRESSURE STEEL BOMB. STEEL BOMB WALLS ARE ONLY CATALYST.

PETROLEUM REFINING LABORATORY
SCHOOL OF CHEMISTRY AND PHYSICS

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JULY 17, 1952

TABLE XV
OXIDATION STUDIES AT 500°F.

PROCEDURES AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-6987.

TEST CONDITIONS: TEST TEMPERATURE = $500 \pm 5^\circ\text{F}$.; TEST TIME = 6 HOURS; AMOUNT OF TEST FLUID AS INDICATED;
AIR RATE = 10 ± 1 LITERS PER HOUR; NO METAL CATALYSTS PRESENT IN SYSTEM.

TEST FLUID DESIGNATION, PRL NO.	3103 DI-2-ETHYL- HEXYL SEBACATE 0.0 0.5		3176 TRIETHYLENE GLYCOL DI-2-ETHYL HEXANOATE 0.5	3057 HERCOFLEX 600 0.5	3107 DI-2-ETHYL- HEXYL PHTHALATE 0.5	2850 DI-BUTOXY- ETHYL SEBACATE 0.5	9900 POLYGLYCOL E900 0.5	9925 SILICONE 701 (DOW CORNING) 0.0
LIQUID CHARGED, GRAMS	90.0	89.0	95.5	98.5	100.0	96.5	110.0	100.0
LIQUID IN TUBE FOLLOWING TEST, GRAMS	82.0	87.0	93.0	96.0	99.5	88.5	109.5	100.0
VOLATILE PRODUCTS IDENTIFIED AS ⁽¹⁾								
CARBON DIOXIDE, GRAMS	2.2	0.6	1.6	1.0	0.3	0.6	0.4	0.1
WATER, GRAMS	1.9	1.3	2.2	0.1	1.0	1.5	0.1	0.5
HYDROCARBON, GRAMS	3.4	0.7	1.5	1.3	0.7	4.7	1.0	0.0
TOTAL VOLATILE PRODUCTS, GRAMS	7.5	2.6	5.3	2.4	2.0	6.8	1.5	0.6
PROPERTIES OF LIQUID PRODUCT FROM TUBE:								
CHANGE IN VISCOSITY AT 130°F.	+21	+13	+6	+27	+2	+18	+4	+11
0°F.	+52	+26	+19	+67	+6	SOLID	SOLID	+26
NEUT. NO. (MG. KOH/GM. OIL):								
ORIGINAL	0.1	0.1	0.1	0.0	0.1	1.6	-0.4	0.0
FINAL	19.6	8.6	8.1	4.2	7.4	19.6	0.1	0.1
ASTM UNION COLOR:								
ORIGINAL	2	2	1	1	1	1	2	1
FINAL	>8	>8	>8	>8	>8	>8	>8	2
WT. % INSOLUBLE MATERIAL	TRACE	TRACE	0.4	TRACE	0.5	TRACE	0.1	TRACE
PROPERTIES OF VOLATILE PRODUCTS:								
WATER LAYER:								
NEUT. NO.	-	50	75	27	119	59	-	-
HYDROCARBON LAYER:								
NEUT. NO.	26	12	148	101	9	35	4	-
BROMINE NO.	85	74	9	3	95	72	-	-

(1) VOLATILE PRODUCTS ARE COLLECTED BY PASSING EXHAUST GASES THROUGH A DRY ICE TRAP, DRIERITE, ASCARITE, AND ACTIVATED CHARCOAL. IT HAS BEEN DETERMINED THAT THE AIR USED DOES NOT CONTAIN ANY APPRECIABLE AMOUNTS OF WATER OR CARBON DIOXIDE.

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THE PENNSYLVANIA STATE COLLEGE
STATE COLLEGE, PENNSYLVANIA
MARCH 10, 1952

TABLE XVI
OXIDATION STUDIES AT 500°F.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = $500 \pm 5^\circ\text{F}$.; TEST TIME = 6 HOURS; AIR RATE = 10 ± 1 LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.;
CATALYST = 3 MM. GLASS BEADS; CATALYST AREA = 1500 CM.²

TEST FLUID	PRL 3103 DI-2- ETHYLHEXYL SEBACATE		PRL 3376 DI-2- ETHYLHEXYL PHTHALATE		PRL 3057 HERCOFLEX 600	PRL 3177 POLYETHYLENE GLYCOL DI-2-ETHYL- HEXANOATE	PRL 3370 DIPROPYLENE GLYCOL DI- PELARGONATE	PRL 2973 VOLTESSO 36
PHENOTHIAZINE, WT. %	NONE	0.5	NONE	0.5	0.5	0.5	0.5	NONE
LIQUID CHARGED, GRAMS	90.5	90.0	95.5	96.5	99.0	94.5	91.0	85.5
LIQUID CHARGED, MOLS	0.21	0.21	0.25	0.25	0.21	0.21	0.22	0.34
LIQUID IN TUBE FOLLOWING TEST, GRAMS	76.0	83.5	78.0	92.5	94.5	78.5	83.0	74.5
% RECOVERY (BASED ON TOTAL CHARGE)	86 (4)	96	90 (4)	96	98	94	99	95
APPROX. AMOUNT OF OXYGEN SUPPLIED, GRAMS ⁽¹⁾	15.7	15.7	15.7	15.7	15.7	15.7	15.7	15.7
APPROX. AMOUNT OF OXYGEN USED, GRAMS ⁽¹⁾	8.8	5.9	7.2	2.8	6.0	7.1	6.4	8.3
APPROX. MOLS OF OXYGEN USED/MOL OF ESTER	1.3	0.9	0.9	0.4	1.0	1.0	0.9	0.8
VOLATILE PRODUCTS IN COLD TRAP								
WATER LAYER: WEIGHT, GRAMS	2.0	3.0	2.0	2.0	3.5	8.0	4.6	6.0
NEUT. NO.	28	66	49	47	67	96	72	73
HYDROCARBON LAYER: WEIGHT, GRAMS	4.8	3.8	8.4	0.8	1.6	5.6	5.6	8.0
NEUT. NO.	11	25	31	16	91	21	44	5.1
BROMINE NO.	68	31	39	51	-	10	5	7.0
VOLATILE PRODUCTS IN ABSORPTION TUBES ⁽²⁾								
CARBON DIOXIDE, GRAMS	2.79	1.79	3.27	0.19	2.47	3.55	2.67	0.87
WATER, GRAMS	0.27	0.47	0.43	0.16	0.06	0.03	0.20	0.17
HYDROCARBON, GRAMS	0.02	0.26	0.63	0.12	0.46	0.24	0.52	0.24
TOTAL VOLATILE PRODUCTS, GRAMS ⁽³⁾	9.9	9.4	14.8	3.3	8.2	17.5	13.6	15.3
PROPERTIES OF LIQUID PRODUCT FROM TUBE								
CHANGE IN VISCOSITY AT								
130°F.	+74	+46	+77	+14	+70	+4	+47	+155
0°F.	+209	+196	+3772	+38	+240	+27	+130	+730
NEUT. NO. (MG. KOH/GRAM OIL):								
ORIGINAL	0.1	0.1	0.3	0.3	0.0	1.5	0.3	0.1
FINAL	26	26	32	18	19	14	16	10
WT. % INSOLUBLE MATERIAL	TRACE	0.3	3.9	0.8	TRACE	TRACE	0.4	TRACE
ASTM UNION COLOR:								
ORIGINAL	2	2	1	2	2	2	1	2
FINAL	>8	>8	>8	>8	>8	>8	>8	>8

- (1) AMOUNT OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR AT S.T.P.) X TIME (HR.) X O₂ CONTENT (FRACTION) X 1.43 (GM./LITER). AMOUNT OF OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O₂. THE AMOUNT OF OXYGEN USED IS PLOTTED AGAINST TIME.
- (2) VOLATILE PRODUCTS ARE COLLECTED BY PASSING EXHAUST GASES THROUGH A DRY ICE TRAP, DRIERITE, ASCARITE, AND ACTIVATED CHARCOAL. IT HAS BEEN DETERMINED THAT THE AIR USED DOES NOT CONTAIN ANY APPRECIABLE AMOUNTS OF WATER OR CARBON DIOXIDE.
- (3) THIS FIGURE IS THE TOTAL VOLATILE PRODUCTS INCLUDING THE LIQUIDS IN THE COLD TRAP AND THE GASES ABSORBED IN THE ABSORPTION TUBES.
- (4) EXCESSIVE LOSS BELIEVED TO BE DUE TO LEAKS IN SYSTEM.

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THE PENNSYLVANIA STATE COLLEGE
STATE COLLEGE, PENNSYLVANIA
NOVEMBER 28, 1952

TABLE XVII
OXIDATION STUDIES AT 500°F. WITH METAL CATALYSTS

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = $500 \pm 5^\circ\text{F}$.; TEST TIME = 6 HOURS; AIR RATE = 10 ± 1 LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; TOTAL CATALYST AREA = 1500 CM.²; CATALYST = 50/50 COPPER-STEEL JACK CHAIN.

TEST FLUID	PRL 3103 DI-2- (3) ETHYLHEXYL SEBACATE	PRL 3045 DI-2- (3) ETHYLHEXYL SEBACATE	PRL 3376 DI-2- ETHYLHEXYL PHTHALATE	PRL 3057 HERCOFLEX 600	PRL 3176 TRIETHYLENE GLYCOL DI-2-ETHYL HEXANOATE	PRL 3177 POLYETHYLENE GLYCOL DI- 2-ETHYL HEXANOATE	PRL 3370 DIPROPYLENE GLYCOL DI- PELARGONATE	PRL 2973 VOLTESO 36
PHENOTHIAZINE, WT.-%	0.5	0.5	0.5	0.5	0.5	0.5	0.5	NONE
LIQUID CHARGED, GRAMS	90.5	90.0	89.5	93.5	95.5	97.5	94.5	85.0
LIQUID CHARGED, MOLS	0.21	0.21	0.21	0.24	0.24	0.21	0.21	0.33
LIQUID IN TUBE FOLLOWING TEST, GRAMS	79.0	87.5	82.0	85.0	88.0	92.5	84.5	75.5
% RECOVERY (BASED ON TOTAL CHARGED)	95	97	98	98	95	98	88.4	97
APPROX. AMOUNT OF OXYGEN SUPPLIED, GMS. (1)	15.7	15.7	15.7	15.7	15.7	15.7	15.7	15.7
APPROX. AMOUNT OF OXYGEN USED, GMS. (1)	9.1	7.0	8.3	5.6	6.4	6.1	5.1	8.5
APPROX. MOLS OF OXYGEN USED/MOL OF ESTER	1.3	1.0	1.2	0.7	0.9	0.9	1.0	0.9
VOLATILE PRODUCTS IN COLD TRAP:								
WATER LAYER: WEIGHT, GRAMS	4.5	3.0	3.0	3.0	2.5	3.5	4.0	6.5
NEUT. NO.	31.2	41	38	62	55	36	33	32
HYDROCARBON LAYER: WEIGHT, GRAMS	4.8	3.6	3.6	4.8	2.4	0.8	3.6	3.4
NEUT. NO.	22	28	22	54	32	181	46	8.1
BROMINE NO.	18	23	16	22	38	2.2	10	7.1
VOLATILE PRODUCTS IN ABSORPTION TUBES: (2)								
CARBON DIOXIDE, GRAMS	4.87	3.7	3.95	3.36	2.20	3.71	5.80	1.80
WATER, GRAMS	0.95	0.46	0.44	0.87	0.05	0.23	0.28	0.09
HYDROCARBON, GRAMS	0.45	0.25	0.15	0.30	0.38	0.23	0.22	0.37
TOTAL VOLATILE PRODUCTS, GRAMS	15.6	12.5	11.1	12.4	9.4	8.5	13.9	17.2
PROPERTIES OF LIQUID FROM TUBE:								
% CHANGE IN VISCOSITY AT 130°F.	+317	+70	+49	+16	+9	+54	+11	SOLID
NEUT. NO. (MGS. KOH/GM. OIL):	+515	+115	+90	+41	+17	+170	+29	SOLID
ORIGINAL	0.1	0.1	0.1	0.3	0.3	0.1	0.8	0.1
FINAL	9.0	11.0	8.9	9.8	10.2	8.3	4.3	-
A.S.T.M. UNION COLOR:								
ORIGINAL	2	2	2	2	2	2	2	2
FINAL	>8	>8	>8	>8	>8	>8	>8	>8
WT.-% INSOLUBLE MATERIAL	1.5	2.5	3.1	3.0	3.7	0.5	1.5	14.2
CHANGE IN WT. OF CATALYST (MG./CM. ²)								
STEEL	+0.25	+0.22	+0.30	+0.32	+0.84	+0.12	+0.24	+0.12
COPPER	-0.20	-0.16	-0.10	-0.46	-0.29	-0.20	-0.29	-0.03

- (1) AMOUNT OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HR.) X O₂ CONTENT (FRACTION) X 1.43 (GM./LITER). AMOUNT OF OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O₂. THE AMOUNT OF OXYGEN USED IS PLOTTED AGAINST TIME.
- (2) VOLATILE PRODUCTS ARE COLLECTED BY PASSING EXHAUST GASES THROUGH A DRY ICE TRAP, DRIERITE, ASCARITE, AND ACTIVATED CHARCOAL. IT HAS BEEN DETERMINED THAT THE AIR USED DOES NOT CONTAIN ANY APPRECIABLE AMOUNTS OF WATER OR CARBON DIOXIDE.
- (3) PRL 3103 IS A PLASTICIZER GRADE ESTER. PRL 3045 IS A DISTILLED GRADE ESTER.
- (4) EXCESSIVE LOSS BELIEVED TO BE DUE TO LEAKS IN SYSTEM.

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THE PENNSYLVANIA STATE COLLEGE
STATE COLLEGE, PENNSYLVANIA
DECEMBER 10, 1952

TABLE XVIII
OXIDATION STUDIES AT 500°F.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = $500 \pm 5^\circ\text{F}$.; TEST TIME = 6 HOURS; AIR RATE = 10 ± 1 LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.;
CATALYST SURFACE = 1500 SQ. CM. (50:50 COPPER:STEEL JACK CHAIN).

TEST FLUID	PRL 3382 SILICATE-BASE FLUID (MLO 5277)	PRL 3416 SILICATE-BASE FLUID (MLO 5277)	PRL 3383 SILICATE-BASE FLUID (MLO 6226)	PRL 3325 O.C. SILICONE OIL	PRL 3358 G.E. SILICONE OIL	PRL 3357 G.E. SILICONE OIL
LIQUID CHARGED, GRAMS	87.5	88.0	88.0	102.0	98.0	98.0
LIQUID CHARGED, MOLES	0.18	0.18	0.24	-	-	-
LIQUID IN TUBE FOLLOWING TEST, GRAMS	73.0	73.0	56.0	102.0	96.5	97.0
% RECOVERY (BASED ON TOTAL CHARGE)	86(3)	95	97	99	98	99
APPROX. AMOUNT OF OXYGEN SUPPLIED, GRAMS ⁽¹⁾	15.7	15.7	15.7	15.7	15.7	15.7
APPROX. AMOUNT OF OXYGEN USED, GRAMS ⁽¹⁾	2.8	3.5	7.1	0.3	0.16	0.16
APPROX. MOLES OF OXYGEN USED/MOLE OF ESTER	0.5	0.6	0.9	-	-	-
VOLATILE PRODUCTS IN COLD TRAP:						
WATER LAYER: WEIGHT, GRAMS	0.5	1.0	2.0	TRACE	TRACE	TRACE
NEUT. NO.	5.9	8.3	14.7	-	-	-
HYDROCARBON LAYER: WEIGHT, GRAMS	3.6	11.6	31.2	TRACE	TRACE	TRACE
NEUT. NO.	7.3	5.7	8.7	-	-	-
BROMINE NO.	5.7	2.2	2.1	-	-	-
VOLATILE PRODUCTS IN ABSORPTION TUBES: ⁽²⁾						
CARBON DIOXIDE, GRAMS	0.76	1.10	1.42	0.13	0.11	0.13
CARBON MONOXIDE, GRAMS	-	0.6	1.7	TRACE	TRACE	TRACE
WATER, GRAMS	0.00	0.00	0.08	0.00	0.04	0.06
HYDROCARBON, GRAMS	0.07	0.02	0.02	0.02	0.00	0.00
TOTAL VOLATILE PRODUCTS, GRAMS	5.0	13.7	36.4	0.15	0.15	0.19
PROPERTIES OF LIQUID FROM TUBE:						
% CHANGE IN VISCOSITY AT						
130°F.	+22	+44	+478	+7	+2	+3
0°F.	+48	+46	+2130	+15	+3	+7
NEUT. NO. (MGS. KOH/GRAM OIL):						
ORIGINAL	0.1	0.1	0.1	0.1	0.1	0.0
FINAL	0.3	0.8	8.0	0.4	0.1	0.0
A.S.T.M. UNION COLOR:						
ORIGINAL	2	2	2	1	1	1
FINAL	>8	>8	>8	2	2	2
WT.% INSOLUBLE MATERIAL	0.5	1.0	0.5	NONE	NONE	NONE
CHANGE IN WT. OF CATALYST (MG./CM. ²)						
STEEL	+0.16	+0.13	+0.08	+0.01	0.00	+0.02
COPPER	+0.05	-0.16	-0.07	+0.04	+0.04	-0.20

- (1) AMOUNT OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HR.) X O₂ CONTENT (FRACTION) X 1.43 (GM./LITER). AMOUNT OF OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O₂. THE AMOUNT OF OXYGEN USED IS PLOTTED AGAINST TIME.
- (2) VOLATILE PRODUCTS ARE COLLECTED BY PASSING EXHAUST GASES THROUGH A DRY ICE TRAP, DRIERITE, ASCARITE, AND ACTIVATED CHARCOAL. IT HAS BEEN DETERMINED THAT THE AIR USED DOES NOT CONTAIN ANY APPRECIABLE AMOUNTS OF WATER OR CARBON DIOXIDE.
- (3) EXCESSIVE LOSS BELIEVED TO BE DUE TO LEAKS IN SYSTEM.
- PETROLEUM REFINING LABORATORY
SCHOOL OF CHEMISTRY AND PHYSICS
- THE PENNSYLVANIA STATE COLLEGE
STATE COLLEGE, PENNSYLVANIA
JANUARY 31, 1953

TABLE XIX

OXIDATION STUDIES AT 500°F. WITH METAL CATALYSTS

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = $500 \pm 5^\circ\text{F}$.; TEST TIME = 6 HOURS; AIR RATE = 10 ± 1 LITERS/HOUR; TEST FLUID CHARGED = 100 ML.; AND CATALYST SURFACE = 1500 SQ. CM. (50:50 COPPER-STEEL JACK CHAIN).

TEST FLUID	PRL 3103 DI-2-ETHYLHEXYL SEBACATE	PRL 3103(3) DI-2-ETHYLHEXYL SEBACATE	PRL 3325 DOW CORNING SILICONE OIL	PRL 3424 50 WT.% DI-2-ETHYLHEXYL SEBACATE + 50 WT.% DOW CORNING SILICONE OIL
PHENOTHIAZINE, WT.%	0.5	0.5	-	0.5
LIQUID CHARGED, GRAMS	90.0	90.0	102.0	95.0
LIQUID CHARGED, MOLES	0.21	0.21	-	-
LIQUID IN TUBE FOLLOWING TEST, GMS.	81.5	81.5	102.0	79.0
% RECOVERY (BASED ON TOTAL CHARGE)	97	94	99	83
APPROX. AMOUNT OF OXYGEN SUPPLIED, GRAMS ⁽¹⁾	15.7	15.7	15.7	15.7
APPROX. AMOUNT OF OXYGEN USED, GRAMS ⁽¹⁾	7.0	6.8	0.3	9.0
APPROX. MOLES OF OXYGEN USED/MOLE OF ESTER	1.0	1.0	-	-
VOLATILE PRODUCTS IN COLD TRAP:				
WATER LAYER: WEIGHT, GRAMS	3.0	2.5	TRACE	2.0
NEUT. NO.	41	39.8	-	37
HYDROCARBON LAYER: WEIGHT, GRAMS	3.6	3.6	TRACE	4.4
NEUT. NO.	28	28.3	-	16
BROMINE NO.	23	12.7	-	-
VOLATILE PRODUCTS IN ABSORPTION TUBES: ⁽²⁾				
CARBON DIOXIDE, GRAMS	3.7	1.53	0.13	0.46
CARBON MONOXIDE, GRAMS	1.4	-	TRACE	-
WATER, GRAMS	0.46	0.80	0.00	0.29
HYDROCARBON, GRAMS	0.25	0.43	0.02	0.40
TOTAL VOLATILE PRODUCTS, GRAMS	12.5	8.9	0.15	7.8
PROPERTIES OF LIQUID FROM TUBE:				
% CHANGE IN VISCOSITY				
AT 130°F.	+70	+22	+7	+41
AT 0°F.	+115	+44	+15	+77
NEUT. NO. (MG. KOH/GM. OIL):				
ORIGINAL	0.1	0.1	0.1	0.1
FINAL	11.0	9.0	0.4	10.5
A.S.T.M. UNION COLOR:				
ORIGINAL	2	-	1	-
FINAL	>8	-	2	-
WT.% INSOLUBLE MATERIAL	2.5	9.5	NONE	5.6
CHANGE IN WT. OF CATALYSTS (MG./CM. ²)				
STEEL	+0.22	+0.23	+0.01	+0.57
COPPER	-0.16	-0.11	+0.04	-0.13

- (1) AMOUNT OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HR.) X O_2 CONTENT (FRACTION) X 1.43 (GM./LITER). AMOUNT OF OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O_2 . THE AMOUNT OF OXYGEN USED IS PLOTTED AGAINST TIME.
- (2) VOLATILE PRODUCTS ARE COLLECTED BY PASSING EXHAUST GASES THROUGH A DRY ICE TRAP, DRIERITE, ASCARITE, AND ACTIVATED CHARCOAL. IT HAS BEEN DETERMINED THAT THE AIR USED DOES NOT CONTAIN ANY APPRECIABLE AMOUNTS OF WATER OR CARBON DIOXIDE.
- (3) THE JACK CHAIN CATALYST USED IN THIS OXIDATION TEST WAS TAKEN FROM A PREVIOUS 500°F. TEST WITH DOW CORNING SILICONE OIL (PRL 3325). THIS CATALYST WAS UNIFORMLY COATED FOLLOWING THE PREVIOUS TEST.

PETROLEUM REFINING LABORATORY
SCHOOL OF CHEMISTRY AND PHYSICS

THE PENNSYLVANIA STATE COLLEGE
STATE COLLEGE, PENNSYLVANIA
FEBRUARY 27, 1953

TABLE XX
OXIDATION AND CORROSION PROPERTIES FOR VARIOUS SYNTHETIC FLUIDS AT 500°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = $500 \pm 5^\circ\text{F}$.; TEST TIME = 24 HOURS; AIR RATE = 0.6 LITERS PER HOUR; TEST FLUID = 100 ML.; AND CATALYST = 1 INCH SQUARES EACH OF COPPER, STEEL, AND ALUMINUM.

TEST FLUID PRL 3161 = A SPEC. MIL-L-7808 TYPE FLUID (PREPARED COMMERCIALY)

PRL 3405 = 9.0 WT.% ACRYLOID HF-25 + 1.0 WT.% PARANOX 441 IN DI-2-ETHYLHEXYL SEBACATE.

TEST FLUID	PRL 3103 DI-2- ETHYL- HEXYL SEBACATE 0.5	- PRL 3161 - BATCH 6981 BATCH 5380	PRL 3405 + 0.5 WT.% DIMETHYL ACID PHOSPHITE -	PRL 3057 HERCOFLEX 600 0.5	PRL 3176 TRIETHYLENE GLYCOL DI-2-ETHYL- HEXANOATE 0.5	PRL 3370 DIPROPYLENE GLYCOL DIPELARGONATE 0.5	PRL 2850 DI- BUTOXY- ETHYL SEBACATE 0.5	PRL 3416 SILICATE BASE HYDRAULIC FLUID -	PRL 3325 SILICONE (D.C.) -
PHENOTHIAZINE, WT.%	0.5	-	-	-	0.5	0.5	0.5	-	-
LIQUID CHARGED, GRAMS	90	100	90	89	97	95	91	95	101.5
LIQUID CHARGED, MOLS	0.21	0.24	0.21	0.21	0.18	0.19	0.22	0.23	-
LIQUID LOSS, WT.%	2	0	1	-	0	3	3	2	1
APPROX. AMOUNT OF O ₂ SUPPLIED, GMS. (1)	3.78	3.78	3.78	3.78	3.78	3.78	3.78	3.78	3.78
APPROX. AMOUNT OF O ₂ USED, GMS. (1)	1.36	1.25	1.10	1.29	0.98	1.51	1.63	0.68	0.38
APPROX. % OF O ₂ USED	36	33	29	34	26	40	43	18	10
MOLS OF O ₂ /MOL OF FLUID	0.21	0.16	0.16	0.19	0.17	0.25	0.23	0.09	-
% CHANGE IN VISCOSITY AT 130°F.	+7	-27	-20	-70	+12	+4	+13	+2	+20
AT 0°F.	+17	-23	-11	-70	+29	+10	+28	-	+38
NEUT. NO. (MG. KOH/GM. OIL)									
ORIGINAL	0.1	0.2	0.1	0.3	0.1	0.0	0.2	1.6	0.1
FINAL	6.0	5.6	8.5	35.6	2.6	4.3	20.7	16.1	0.1
WT.% INSOLUBLE MATERIAL	0.1	TRACE	TRACE	0.3	TRACE	0.4	0.1	TRACE	NONE
FINAL CATALYST CONDITION									
APPEARANCE									
COPPER	CORRODED	DULL	CORRODED	DULL	DULL	DULL	DULL	DULL	DULL
STEEL	DULL	DULL	DULL	COATED	DULL	DULL	CORRODED	DULL	DULL
ALUMINUM	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL
WT. (MG./SQ. CM.)									
COPPER	0.25	0.12	0.42	0.14	0.04	0.05	0.06	0.00	0.12
STEEL	+0.02	0.02	+0.07	+0.56	-0.01	+0.05	0.61	+0.02	0.02
ALUMINUM	0.01	0.01	0.00	+0.10	0.00	+0.02	0.04	+0.01	0.02

(1) AMOUNT OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HR.) X O₂ CONTENT (FRACTION) X 1.43 (GM./LITER). AMOUNT OF OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O₂.

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MAY 13, 1953

TABLE XXI
OXIDATION AND CORROSION PROPERTIES FOR VARIOUS FLUIDS AT 500°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-6387.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = $500 \pm 5^\circ\text{F}$.; TEST TIME = 24 HOURS; AIR RATE = 0.6 LITER/HOUR; TEST FLUID = 100 ML.; AND CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, AND ALUMINUM.

TEST FLUIDS: THE FOLLOWING TEST	PRL DESIG.	FLUID	APPROX. CS. VISC. AT 100°F.
FLUIDS ARE ALL LOW POUR MINERAL	PRL 3134	VOLTESSO 36	9
OIL FRACTIONS HAVING VISCOSITY	PRL 2053	NECTON 42	32
LEVELS SHOWN.	PRL 2782	NECTON 45	45

TEST FLUID	PRL 3439 INDOPOL POLYBUTENE L-10(1)	PRL 3440 INDIPOL POLYBUTENE L-50(1)	PRL 3134 VOLTESSO 36	PRL 2053 NECTON 42	PRL 2782 NECTON 45	PRL 2782, NECTON 45 (SILICA GEL TREATED) ⁽²⁾ CUT NO. 1	CUT NO. 3
OXIDATION INHIBITOR, WT.%	-	-	-	-	- 1.0 PHENYL ALPHA NAPHTHYLAMINE	-	-
LIQUID CHARGED, GRAMS	81	79	84	86	87	87	83
LIQUID LOSS, WT.%	3	15	1	2	0	0	2
APPROX. AMOUNT OF O ₂ SUPPLIED, GMS. ⁽³⁾	3.78	3.78	3.78	3.78	3.78	3.78	3.78
APPROX. AMOUNT OF O ₂ USED, GMS. ⁽³⁾	2.27	1.93	2.88	1.70	2.04	0.87	2.01
APPROX. % OF O ₂ USED	60	51	76	45	54	23	53
MOLS OF O ₂ /426 GMS. OF OIL ⁽⁴⁾	0.38	0.33	0.45	0.26	0.31	0.13	0.32
% CHANGE IN VISCOSITY							
AT 130°F.	+15	+121	+15	+19	+12	+11	+10
AT 0°F.	+69	+553	+48	-	-	-	-
NEUT. NO. (MG. KOH/GM. OIL)							
ORIGINAL	0.0	0.0	0.0	0.1	0.1	0.1	0.0
FINAL	0.6	0.7	1.6	1.7	1.1	0.4	1.5
WT. % INSOLUBLE MATERIAL	TRACE	TRACE	TRACE	TRACE	TRACE	TRACE	TRACE
FINAL CATALYST CONDITION							
APPEARANCE							
COPPER	DULL	DULL	DULL	COATED	COATED	COATED	BRIGHT
STEEL	DULL	DULL	DULL	DULL	DULL	DULL	DULL
ALUMINUM	DULL	BRIGHT	DULL	BRIGHT	DULL	DULL	BRIGHT
WT. LOSS (MG./SQ. CM.)							
COPPER	+0.02	+0.02	+0.02	+0.12	+0.10	+0.10	0.00
STEEL	0.02	+0.02	0.02	0.00	0.02	+0.02	0.00
ALUMINUM	0.02	0.01	0.01	0.00	0.00	0.00	0.00

(1) THIS FLUID WAS OBTAINED FROM THE INDOIL CHEMICAL COMPANY, CHICAGO, ILLINOIS.

(2) THE OIL WAS PERCOLATED THROUGH A SILICA GEL COLUMN.

(3) AMOUNT OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HR.) X O₂ CONTENT (FRACTION) X 1.43 (GM./LITER). AMOUNT OF OXYGEN USED IS DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O₂.

(4) THE MOLS OF O₂ FOR 426 GRAMS OF OIL HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE AMOUNT OF O₂ ABSORBED. THE MOLECULAR WEIGHT OF DI-2-ETHYLHEXYL SEBACATE IS 426.

(CONCLUDED ON NEXT PAGE)

TABLE XXI (Concluded)
OXIDATION AND CORROSION PROPERTIES FOR VARIOUS FLUIDS AT 500°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-6387.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = $500 \pm 5^\circ\text{F}$.; TEST TIME = 24 HOURS; AIR RATE = 0.6 LITER/HOUR; TEST FLUID = 100 ML.; AND CATALYST = A
1 INCH SQUARE EACH OF COPPER, STEEL, AND ALUMINUM.

TEST FLUIDS: THE FOLLOWING TEST FLUIDS ARE ALL LOW POUR MINERAL OIL FRACTIONS HAVING VISCOSITY LEVELS SHOWN.

	PRL DESIG.	FLUID			APPROX. CS. VISC. AT 100°F.		
	PRL 2054	NECTON 55			98		
	PRL 3468	HUMBLE PALE D			200		
TEST FLUID	PRL 2054				20 WT.% POLY-BUTENE 8-12 IN NECTON 55	PRL 3468	
	NECTON 55					HUMBLE PALE D	
OXIDATION INHIBITOR, WT.%	-	1.0 PHENYL ALPHA NAPHTHYLAMINE	1.0 PHENYL ALPHA NAPHTHYLAMINE	1.0 ANTI-OXIDANT 2246	-	-	1.0 PHENYL ALPHA NAPHTHYLAMINE
LUBRICATION ADDITIVE, WT.%	-	-	0.5 DIBUTYL ACID PHOSPHITE	0.5 DIBUTYL ACID PHOSPHITE	-	-	-
LIQUID CHARGED, GRAMS	84	85	86	86	82	89	88
LIQUID LOSS, WT.%	1	1	1	1	0	2	0
APPROX. AMOUNT OF O ₂ SUPPLIED, GMS.(3)	3.78	3.78	3.78	3.78	3.78	3.78	3.78
APPROX. AMOUNT OF O ₂ USED, GMS.(3)	1.63	1.04	1.48	1.25	1.26	1.78	1.17
APPROX. % OF O ₂ USED	43	27	39	33	33	47	31
MOLS OF O ₂ /426 GMS. OF OIL(4)	0.25	0.16	0.23	0.19	0.20	0.27	0.18
% CHANGE IN VISCOSITY AT 130°F.	+10	+11	+15	+14	-72	+21	+23
AT 0°F.	-	-	-	-	-	-	-
NEUT. NO. (MG. KOH/GM. OIL)							
ORIGINAL	0.0	0.0	0.2	0.1	0.0	0.0	0.1
FINAL	1.2	0.4	1.9	0.4	1.1	1.1	0.6
WT.% INSOLUBLE MATERIAL	0.1	TRACE	0.1	0.7	TRACE	TRACE	0.1
FINAL CATALYST CONDITION							
APPEARANCE							
COPPER	COATED	COATED	COATED	DULL	COATED	COATED	COATED
STEEL	DULL	DULL	COATED	COATED	DULL	DULL	DULL
ALUMINUM	DULL	BRIGHT	COATED	COATED	BRIGHT	DULL	DULL
WT. LOSS (MG./SQ. CM.)							
COPPER	+0.12	+0.16	+0.07	0.05	+0.05	+0.17	+0.22
STEEL	0.03	0.00	+0.37	+0.57	0.05	0.03	+0.03
ALUMINUM	0.00	0.00	+0.40	+0.62	0.01	0.00	0.00

(3) AMOUNT OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HR.) X O_2 CONTENT (FRACTION) X 1.43 (GM./LITER). AMOUNT OF OXYGEN USED IS DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O_2 .

(4) THE MOLS OF O_2 FOR 426 GRAMS OF OIL HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE AMOUNT OF O_2 ABSORBED. THE MOLECULAR WEIGHT OF DI-2-ETHYLHEXYL SEBACATE IS 426.

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TABLE XXII
OXIDATION AND CORROSION PROPERTIES FOR SEVERAL FLUIDS AT 500°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-6387.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = $500 \pm 5^\circ\text{F}$.; AIR RATE = 0.6 LITERS/HOUR; TEST TIME = 6 HOURS; FLUID CHARGED = 100 ML.; AND CATALYST = 750 SQUARE CENTIMETERS EACH OF COPPER AND STEEL JACK CHAIN.

FLUIDS NECTON 45 AND NECTON 55 ARE LOW POUR MINERAL OIL FRACTIONS HAVING VISCOSITIES OF 45 AND 98 CENTISTOKES, RESPECTIVELY.

TEST FLUID	PRL 3103 DI-2-ETHYLHEXYL SEBACATE 0.5 PHENO- THIAZINE	PRL 3057 HERCOFLEX 600 0.5 PHENO- THIAZINE	-PRL 2782, NECTON 45- - 1.0 PHENYL ALPHA NAPHTHYLAMINE		- PRL 2054, NECTON 55 - - 1.0 PHENYL ALPHA NAPHTHYLAMINE	
LIQUID CHARGED, GRAMS	90	96	87	87	84	85
LIQUID LOSS, WT. %	4	0	0	1	2	0
APPROX. AMOUNT OF O_2 SUPPLIED, GMS. (1)	0.95	0.95	0.95	0.95	0.95	0.95
APPROX. AMOUNT OF O_2 USED, GMS. (1)	0.51	0.58	0.88	0.63	0.79	0.58
APPROX. % OF O_2 USED	54	62	93	67	84	61
MOLS OF O_2 USED/426 GRAMS OF FLUID (2)	0.08	0.08	0.13	0.10	0.13	0.09
% CHANGE IN VISCOSITY						
AT 130°F .	+6	+6	+6	+7	+5	+8
AT 0°F .	+15	+16	-	-	-	-
NEUT. NUMBER (MG. KOH/GM. OF OIL)						
ORIGINAL	0.1	0.1	0.1	0.1	0.0	0.0
FINAL	9.1	3.4	0.6	0.2	0.4	0.2
WT. % INSOLUBLE MATERIAL	TRACE	TRACE	TRACE	TRACE	TRACE	TRACE
FINAL CATALYST CONDITION						
APPEARANCE						
COPPER	DULL	DULL	DULL	DULL	DULL	DULL
STEEL	DULL	DULL	DULL	DULL	DULL	DULL
WT. LOSS (MG./SQ. CM.)						
COPPER	0.02	0.03	0.00	0.00	0.00	+0.03
STEEL	+0.05	+0.04	+0.02	+0.03	+0.01	+0.03

(1) AMOUNT OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HR.) X O_2 CONTENT (FRACTION) X 1.43 (GM./LITER). AMOUNT OF OXYGEN USED IS DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O_2 .

(2) THE MOLS OF O_2 FOR 426 GRAMS OF OIL HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE AMOUNT OF O_2 ABSORBED. THE MOLECULAR WEIGHT OF DI-2-ETHYLHEXYL SEBACATE IS 426.

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TABLE XXIII
OXIDATION STUDIES WITH DI-2-ETHYLHEXYL SEBACATE AT 500°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-6387.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = $500 \pm 5^\circ\text{F}$.; TEST TIME AS INDICATED; AIR RATE = 5 ± 0.5 LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; AND CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, AND ALUMINUM.

TEST FLUID TEST TIME, HOURS	- - - 0.5 WT.% PHENOTHIAZINE IN DI-2-ETHYLHEXYL SEBACATE (PRL 3207) - - -				
	12	20	30	40	60
LIQUID CHARGED, GRAMS	90.0	90.5	90.0	90.0	91.0
LIQUID LOSS, WT.%	1	7	9	5	0
APPROXIMATE AMOUNT OF O_2 SUPPLIED, GMS. (1)	15.7	26.2	39.3	52.4	78.6
APPROXIMATE AMOUNT OF O_2 USED, GMS. (1)	3.5	5.8	8.7	11.6	17.4
MOLS O_2 USED/MOL OF ESTER (426 GRAMS)	0.52	0.86	1.30	1.72	2.60
% CHANGE IN VISCOSITY					
AFTER REMOVAL OF OIL INSOLUBLE MATERIAL (2)					
AT 130°F .	+18	+44	+116	+164	SOLID (4)
AT 0°F .	+40	+48	+240	+328	SOLID (4)
AFTER REMOVAL OF ISOPENTANE INSOLUBLE MATERIAL (3)					
AT 130°F .	+1	+4	+51	+45	+50
NEUT. NO. (MG. KOH/GM. OIL)					
ORIGINAL	0.1	0.1	0.1	0.1	0.1
FINAL	8.4	15.4	16.8	17.1	36.2
WT.% OIL INSOLUBLE MATERIAL (2)	TRACE	0.1	0.8	0.9	-(4)
WT.% ISOPENTANE INSOLUBLE MATERIAL (3)	1.4	4.8	6.9	8.7	25
FINAL CATALYST CONDITION					
APPEARANCE					
COPPER	DULL	DULL	DULL	CORRODED	CORRODED
STEEL	DULL	DULL	COATED	DULL	CORRODED
ALUMINUM	BRIGHT	BRIGHT	BRIGHT	BRIGHT	BRIGHT
WT. LOSS (MG./SQ. CM.)					
COPPER	0.08	0.10	0.07	0.75	0.52
STEEL	0.00	0.07	+0.23	0.09	3.26
ALUMINUM	0.00	0.00	0.00	0.00	0.03

(1) AMOUNT OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HR.) X O_2 CONTENT (FRACTION) X 1.43 (GM./LITER). AMOUNT OF OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O_2 .

(2) OIL INSOLUBLE MATERIAL IS REMOVED BY CENTRIFUGING OXIDIZED FLUID IMMEDIATELY AFTER COMPLETION OF TEST. THE OIL INSOLUBLES ARE WASHED WITH A LOW BOILING PETROLEUM NAPHTHA AND DRIED BEFORE WEIGHING.

(3) THE ISOPENTANE INSOLUBLE MATERIAL IS REPORTED AS THE SUM OF THE OIL INSOLUBLE MATERIAL PLUS THE ISOPENTANE INSOLUBLE MATERIAL. ISOPENTANE INSOLUBLE MATERIAL IS DETERMINED BY DISSOLVING A SAMPLE OF OXIDIZED FLUID (FROM WHICH THE OIL INSOLUBLES HAVE BEEN REMOVED) IN ISOPENTANE AND WEIGHING THE INSOLUBLE MATERIAL WHICH SETTLES OUT.

(4) OXIDIZED FLUID WAS AN INTIMATE MIXTURE OF INSOLUBLE MATERIAL AND OXIDIZED LIQUID. OIL INSOLUBLES COULD NOT BE DETERMINED WITHOUT DILUTION.

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TABLE XXIV
OXIDATION STUDIES WITH A MINERAL OIL FRACTION AT 500°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-6987.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = $500 \pm 5^\circ\text{F}$.; TEST TIME AS INDICATED; AIR RATE = 5 ± 0.5 LITERS PER HOUR; TEST FLUID = 100 ML.; AND CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, AND ALUMINUM.

TEST FLUID PRL 2782 = NECTON 45, A NAPHTHENIC MINERAL OIL OF 44.6 CENTISTOKES VISCOSITY AT 100°F . OBTAINED FROM THE STANDARD OIL COMPANY OF NEW JERSEY.

TEST FLUID	-- PRL 2782 --				
TEST TIME, HOURS	6	20	30	40	50
LIQUID CHARGED, GRAMS	89.0	90.0	88.0	89.0	89.0
LIQUID LOSS, WT. %	0	2	3	3	8
APPROXIMATE AMOUNT OF O_2 SUPPLIED, GMS. (1)	7.9	26.2	39.3	52.4	65.5
APPROXIMATE AMOUNT OF O_2 USED, GRAMS (1)	1.6	5.2	7.9	10.5	13.1
GRAMS O_2 USED/GRAM OF OIL	0.02	0.06	0.09	0.12	0.15
MOLS O_2 USED/426 GRAMS OF OIL (6)	0.24	0.77	1.20	1.57	1.95
% CHANGE IN VISCOSITY					
AFTER REMOVAL OF OIL INSOLUBLE MATERIAL (2)					
AT 130°F .	+19	+86	+568	SOLID (4)	SOLID (4)
AFTER REMOVAL OF ISOPENTANE INSOLUBLE MATERIAL (3)					
AT 130°F .	+15	+5	-2	-	-
NEUT. NO. (MG. KOH/GM. OIL)					
ORIGINAL	0.1	0.1	0.1	0.1	0.1
FINAL	1.0	2.8	4.9	2.7	3.7
WT. % OIL INSOLUBLE MATERIAL (2)	TRACE	0.3	5.0		
WT. % ISOPENTANE INSOLUBLE MATERIAL (3)	0.4	4.6	8.5	49 (5)	64 (5)
FINAL CATALYST CONDITION					
APPEARANCE					
COPPER	DULL	DULL	COATED	COATED	COATED
STEEL	DULL	DULL	DULL	DULL	DULL
ALUMINUM	BRIGHT	BRIGHT	BRIGHT	DULL	DULL
WT. LOSS (MG./SQ. CM.)					
COPPER	0.00	0.13	+0.12	+0.12	+0.16
STEEL	0.05	+0.02	0.01	0.00	0.00
ALUMINUM	0.00	0.00	0.00	0.00	0.02

(1) AMOUNT OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HR.) X O_2 CONTENT (FRACTION) X 1.43 (GM./LITER). AMOUNT OF OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O_2 .

(2) OIL INSOLUBLE MATERIAL IS REMOVED BY CENTRIFUGING OXIDIZED FLUID IMMEDIATELY AFTER COMPLETION OF TEST. THE OIL INSOLUBLES ARE WASHED WITH A LOW BOILING PETROLEUM NAPHTHA AND DRIED BEFORE WEIGHING.

(3) THE ISOPENTANE INSOLUBLE MATERIAL IS REPORTED AS THE SUM OF THE OIL INSOLUBLE MATERIAL PLUS THE ISOPENTANE INSOLUBLE MATERIAL. ISOPENTANE INSOLUBLE MATERIAL IS DETERMINED BY DISSOLVING A SAMPLE OF OXIDIZED FLUID (FROM WHICH THE OIL INSOLUBLES HAVE BEEN REMOVED) IN ISOPENTANE AND WEIGHING THE INSOLUBLE MATERIAL WHICH SETTLES OUT.

(4) OXIDIZED FLUID WAS AN INTIMATE MIXTURE OF INSOLUBLE MATERIAL AND OXIDIZED LIQUID. OIL INSOLUBLES COULD NOT BE DETERMINED WITHOUT DILUTION.

(5) THESE VALUES ARE WEIGHT PER CENT NAPHTHA INSOLUBLE MATERIAL VALUES DETERMINED BY DILUTING THE OXIDIZED FLUID WITH SUCCESSIVE PORTIONS OF PETROLEUM NAPHTHA AND THEN CENTRIFUGING.

(6) THE MOLS OF O_2 FOR 426 GRAMS OF OIL HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE O_2 ABSORBED. THE MOLECULAR WEIGHT OF DI-2-ETHYL-HEXYL SEBACATE IS 426.

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JUNE 11, 1953

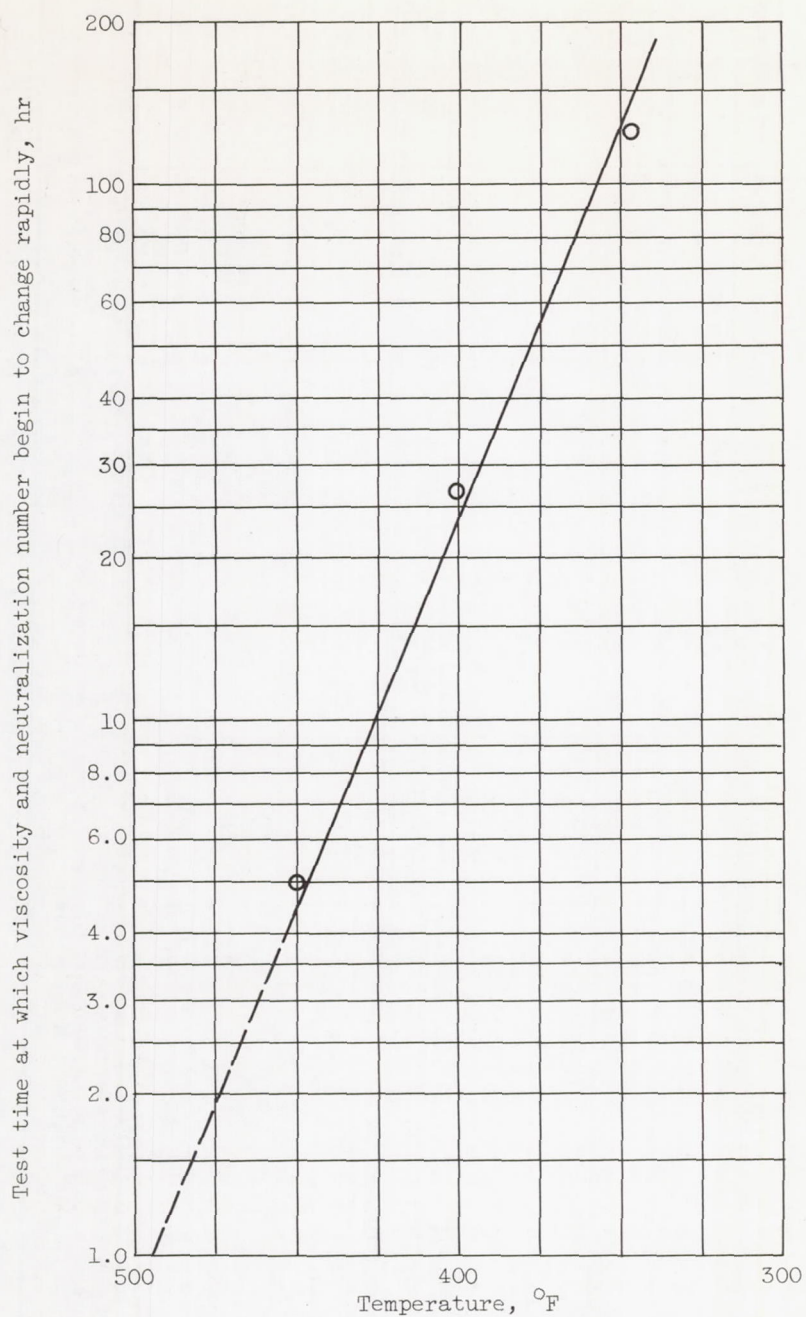


Figure 1. - Effect of temperature on oxidation stability of di(2-ethylhexyl) sebacate (PRL 3371). Technique and procedure in accordance with MIL-L-7808. Air rate, 10 ± 1 liters per hour; test fluid charged, 100 milliliters; metal catalysts as specified. Test fluid, 0.5 percent by weight phenothiazine in di(2-ethylhexyl) sebacate.

APPENDIX F

NACA RESEARCH ON LUBRICANTS, BEARINGS, AND LUBRICATION
FOR HIGH-TEMPERATURE TURBINE ENGINESBy R. L. Johnson and E. E. Bisson
NACA Lewis Laboratory

INTRODUCTION

Since many of the previous papers have discussed in considerable detail the specific problems arising from engine operation at high temperatures, these problems will not be discussed further. Other discussion of problems and turbine-engine requirements with respect to both bearings and lubricants are covered in references 1 and 2, respectively.

The objective of this paper is to discuss briefly some of the NACA research on various problems and possible solutions. It should be noted that the problems with respect to a short-time engine (such as an expendable engine for a guided missile) may be considerably different from the problems for a long-time engine (such as an engine designed for long life in aircraft applications).

LUBRICANTS

Liquids

Miscellaneous properties. - The miscellaneous properties (such as viscosity-temperature, high-temperature stability, corrosivity, and toxicity) have been discussed to some length in other papers and hence will not be covered herein. Some of the properties of the fluids included in the NACA studies are, however, shown in table I.

Lubricating effectiveness at high temperatures. - Studies were made of the lubricating effectiveness at high bulk lubricant temperatures of the various fluids of table I. These studies are described in detail in reference 3. These studies were made on an apparatus (fully described in ref. 4) shown schematically in figure 1. The specimens consist of a rotating steel disk (hardness, Rockwell A-50) and a spherical-tipped steel rider (hardness, Rockwell C-60).

Dead-weight loading is used to apply the load through the pulley system shown in figure 1. The load used in this investigation was 1000 grams (initial Hertz surface stress, 149,000 psi). Friction force was measured by means of four strain gages mounted on a copper-beryllium dynamometer ring.

The experimental procedure consisted in starting each test with a new set of friction specimens and 440 cubic centimeters of fresh fluid. The apparatus was started simultaneously with the application of heat, and friction readings were taken at 45° F intervals, or more often if necessary. The average time required to raise the temperature of the fluid from 100° to 500° F was 55 minutes. Lubrication failure was easily determined during the experiment as an increase in friction force, onset of instability in the friction readings, or audible chatter of the sliding specimens; these points are indicated on friction plots to be discussed. After the runs in which failure occurred, visible surface damage was observed. Preliminary wear tests conducted with some of these showed that either unstable friction values or an increase in the coefficient of friction was accompanied by a sharp increase in the rate of wear.

During the course of each experiment the following observations were considered most important:

- (1) Onset of instability or a marked increase in the friction force.
- (2) Surface failure (incipient or mass) of the sliding specimens.
- (3) Thermal instability of the bulk lubricant.

In figure 2 are shown photomicrographs of surfaces which are considered typical of those obtained with: (a) effective lubrication, (b) incipient failure, and (c) mass failure. The specimens show, respectively: (a) no evidence of surface distress, (b) slight evidence of surface welding and metal transfer, and (c) violent welding and metal transfer. Interpretation of these results is based on the knowledge that, because of violent agitation, there is considerable entrained air in the lubricants, both in these experiments and in actual turbine engines.

Preliminary wear tests, conducted with some of these lubricants, showed that either unstable friction values or an increase in the coefficient of friction was accompanied by a sharp increase in the rate of wear. Figure 3 presents data from one of a series of experiments designed to correlate wear (qualitative) with friction instability as it was encountered in most of the experiments with increasing bulk lubricant temperatures. A dial indicator was attached to the loading system in such a manner that it would measure displacement in the rider holder assembly that resulted from wear of the rider. On the basis of incremental displacement values obtained during an experiment, cumulative values of wear volume were calculated. These wear volumes are indicated in the wear curve of figure 3. No significance should be attached to absolute wear values; the trend of the curve was, however, reproducible and indicates that there is a definite relation between instability or a marked increase in friction and a marked change in the rate of wear. The preliminary data of figure 3 were obtained with a heating rate approximately one-half that utilized in the rest of the experiments reported herein in order to accentuate the wear data.

The results showing the effect of temperature on friction of steel specimens boundary lubricated by the various synthetic lubricants of table I are shown in figure 4. On each of the individual curves of figure 4, the regions of effective boundary lubrication, incipient surface failure, and mass surface failure are shown. The petroleum lubricant grade 1010 (fig. 4(a)) is included for purposes of comparison. The compounded diester of figure 4(b) is essentially a lubricant of the MIL-L-7808 specification type. This lubricant does not quite meet these specifications, however, because of slightly excessive viscosity at -65° F (16,000 cs measured as compared to 13,000 centistokes specification max.). The performance of the base stock, di-2-ethylhexyl sebacate, is shown in figure 5(a). As shown in a comparison of figure 4(b) and figure 5(a), the compounded diester shows appreciably better high-temperature performance than the base stock alone. The increased lubricating effectiveness of the compounded diester over that of the base stock at higher temperatures is shown in figure 5 to be a function of the lubrication or anti-wear additive tricresyl phosphate (fig. 5(c)) and is not dependent on the presence of the oxidation inhibitor (fig. 5(b)) or the viscosity-index improver (fig. 5(d)). The curves of figure 5 show that instability starts at the same temperature for all except the solution of tricresyl phosphate in di-2-ethylhexyl sebacate.

Comparison of the results (fig. 4) of the various fluids of this investigation shows that the phosphonate and the silicate appear to be promising for high-temperature operation. The specimens run with phosphonate showed no surface distress

throughout the entire temperature range (to 572° F with one phosphonate ester). At this temperature, however, the fluid refluxed violently, decomposed, and left a reddish brown gel as a residue. The wear track showed no damage and very slight wear but the surface of the steel specimen appeared to be etched as if by acid wherever contact with the fluid had taken place. Chemical attack of the surfaces at high temperatures is apparently a logical explanation for the lubricating effectiveness of this fluid, since visual inspection of the different specimens operated in two phosphonate esters showed that considerable etching had occurred (ref. 3).

As previously mentioned, consideration of a short-time engine (for example, an expendable engine for guided missile application) would make it appear that the phosphonate esters might be promising for relatively high-temperature operation in spite of the fact that the fluids do cause corrosion. It is possible that the disadvantage of corrosion may be overbalanced by the ability to lubricate effectively at high temperatures, particularly since the corrosive action would take place for such a short period of time.

The tetra(2-ethylhexyl) silicate ester also appears promising for operation at high temperatures. The silicate ester is, however, unstable at bulk temperatures of approximately 540° F. As a consequence, the NACA studies were limited to temperatures of the order of 510° F. As reported in reference 3, the presence of an oxidation inhibitor (phenyl- α -naphthylamine) appeared to have no effect on the lubricating effectiveness of the silicate ester. The oxidation inhibitor also appeared to have no effect on the high-temperature stability of the fluids at temperatures of approximately 540° F. There was no visible evidence of etching of the steel specimens used in the experiments with the silicate esters. At this time there is no explanation for the lubricating effectiveness of the silicate esters at high temperatures.

The data of figure 4(f) are for an NACA silicone-diester blend. Because of the very good viscometric properties of the silicone-diester blends it is believed these fluids will find particular application as turboprop power-reduction-gear lubricants.

Unreported ball bearing experiments have demonstrated that by use of a drop lubrication method in the presence of air, silicone-diester blends will provide effective lubrication at temperatures higher than those at which a compounded diester fluid failed to lubricate. The effectiveness of the silicone-diester blend as a lubricant for high-temperature bearings may be associated with the formation of localized decomposition products such as lacquer on the lubricated surfaces. There will be further discussion of the performance of silicone-diester blends as gear lubricants in a section on lubrication methods for gears.

The frictional results for a series of water-soluble polyalkylene glycol fluids that vary in viscosity from 55 to 3520 SUS (8.9 to 762 centistokes) at 100° F are shown in figure 6. The results obtained at the high temperatures show a definite relation to some property commonly associated with viscosity grade, since increasing the viscosity is shown to result in an increase in the region of effective boundary lubrication. For example, incipient surface failure starts at 290° F for the 55-SUS fluid, whereas the 3520-SUS fluid shows incipient surface failure starting at 501° F.

It should be pointed out that the maximum bearing temperatures for effective lubrication with these fluids may be substantially above the bulk oil temperature limits mentioned herein. In bearing lubrication, the oil is normally supplied at low bulk temperatures and the oil residence time may not be sufficient so that

high bulk oil temperatures are obtained. In some bearing experiments, satisfactory lubrication has been obtained with bearing temperatures approximately 200° F higher than the bulk lubricant failure temperature reported herein.

Lubricating effectiveness at high sliding velocities. - Studies reported in reference 5 of the lubricating effectiveness of many of these fluids at high sliding velocities showed that, with some exceptions, there is a general correlation between the limiting temperature for effective boundary lubrication and the limiting sliding velocity for effective boundary lubrication. That is, those fluids which showed high limiting temperatures also, in general, showed high limiting sliding velocities.

Solids and Gases

Since liquids are inherently temperature-limited, it is possible that there may be considerable promise in the use of either solid or gaseous lubricants for the extreme high-temperature application. Solids can be obtained which are either stable at the elevated temperatures or whose instability does not adversely affect the frictional properties of the surfaces to be lubricated. There will be more discussion of these two lubricant types in a subsequent section on lubrication methods for bearings.

Temperature Ranges for Various Lubricant Types

A summary of the possible lubricants for operation over the four temperature ranges is shown in table II. As shown, petroleum lubricants of the MIL-O-6081A type are suitable for temperature range 1; synthetic lubricants (presently, diesters) of the MIL-L-7808 type are suitable for temperature range 2; solids, gases, or liquids (possible future synthetics or liquids in closed systems) may be suitable for temperature range 3 and, in all probability, only solids and gases will be suitable for the extreme conditions of temperature range 4. Solid lubricants such as MoS₂ or graphite, may be utilized in an airmist system where the solid lubricant is suspended in a fluid such as air and continuously supplied to the bearings at a very slow rate (of the order of ounces per hour). As indicated in table II, additional research is required on high-temperature stability and high-temperature lubricating effectiveness of lubricants for temperature ranges 2, 3, and 4.

BEARINGS

Materials for Races and Rolling Elements

Miscellaneous physical properties. - The physical properties and requirements of bearing materials for operation at high temperatures have been adequately discussed in this conference and in a paper by Frank Wellons which is included in reference 1. Based on the requirements, the various molybdenum tool steels appear to show considerable promise as high-temperature bearing materials. These steels have good hot hardness and dimensional stability at elevated temperature, although they do require considerably longer grinding times than SAE 52100 steel.

Fatigue properties. - Since, as is shown in reference 1, the anticipated requirements for bearings for future high-speed, high-temperature engines involve high thrust loads, high DN values, and high temperatures, it is obvious that the fatigue life of the bearing material must be adequate to permit operation of these bearings under these conditions over extended periods of time. All the requirements tend to make the fatigue problem more difficult and it is possible that the fatigue life of bearings may become a very definite limitation to the life of the engine.

As a consequence, NACA has initiated fatigue studies in an apparatus designed by E. F. Macks and described fully in reference 6. The fatigue spin rig is shown in figure 7. Essentially the apparatus consists of a raceway A which is resiliently mounted in the horizontal plane on toothpick springs B, a vertically adjustable nozzle assembly C, and a vertically adjustable bottom plate D. Pressurized air, entering at E, causes two or more balls to rotate within the raceway. The spent air is allowed to escape at F. The speed of ball rotation is controlled by adjusting the pressure of the air entering at E.

Under proper conditions of operation, the balls lift from the bottom plate to ride hour after hour at an equilibrium position on a vertical raceway wall. Acceleration, gyroscopic, and aerodynamic forces apparently provide the lifting and required stabilization. An analysis of this phenomenon was unsuccessful because of the unknown aerodynamic forces.

Inasmuch as a pair of balls, or a trio of balls of proper mass ratio, rotate in dynamic equilibrium, since the balls do not touch any surface except the raceway wall, the loads at the respective contact areas between the balls and the cylinder may be determined with precision. These loads are caused by the centrifugal forces resulting from the orbital motion of the balls about the raceway axis.

Rolling motion under elastic deformation occurs between the surfaces in contact. A representative ball path obtained in the fatigue spin rig is shown in figure 8. The ball path shown was obtained by rotating a pair of 0.5-inch-diameter chrome steel balls at an orbital speed of 28,000 rpm within a 3.310-inch-bore, ungrooved tool-steel raceway for about 7 minutes, which resulted in 1,358,000 ball cycles. No lubrication was provided. The primary rolling lines and sliding contact bands associated with rolling under elastic deformation are clearly visible. Similar contact bands occur on the raceway. The width of the complete contact band as calculated (0.038 inch, approx.) checks favorably with experimental findings (0.041 inch, approx.), the experimental values being about 8 percent higher.

The stresses produced in the subject apparatus bear the same relation to load as do the stresses in a loaded ball bearing. Surface compressive and tangential stresses and subsurface shear stresses are imposed at the contact area and within the stressed volume, respectively.

Stress frequencies to over 10 million cycles per hour and stress magnitudes to over 700,000 pounds per square inch (calculated, Hertz) have been obtained. For the path shown in figure 8, the maximum calculated surface pressure was 735,000 pounds per square inch. The magnitudes of the imposed stresses are varied by adjustment of the drive-air pressure to change the speed of ball rotation. The stresses can be varied at a given frequency of application by changing specimen geometry. The apparatus can also be operated with high ambient temperature in order to determine fatigue life at elevated temperatures.

Experiments in progress at the present time are designed to compare the fatigue life of M-1 molybdenum tool steel to that of SAE 52100 steel at a temperature of 225° F. Additional studies are being made of: (1) the fatigue life of M-1 tool steel at 450° F and (2) the effect of nonhomogeneity of the material (such as grain direction in the ball as a result of the processing methods).

Materials for Cages

Physical properties and requirements. - As shown in reference 1, the requirements for cage materials are as follows:

- (1) Adequate strength at low and at high temperatures (present cage materials seem to be adequate, at low temperatures only, in this regard)
- (2) Corrosion resistance
- (3) Compatibility
- (4) Thermal expansion coefficient approaching that for the race material

Of these requirements, that of compatibility is probably the most important and possibly the most difficult to obtain practically. It is believed the friction and wear problem will become more acute at high temperatures because of the obvious effect of high temperatures in reducing effective lubrication.

An additional factor of importance (to be discussed in the section "Cage and Bearing Designs") is that of friction coefficient. (The studies of reference 8 show that high friction coefficient can be detrimental to bearing performance by inducing cage slip.) The appearance of cage breakage in high-temperature bearing runs (to be discussed in a section on lubrication methods for bearings) indicates that cage strength can be very important under critical operating conditions.

Friction, welding, and wear properties. - As a consequence of the importance of the compatibility problem, an investigation at high temperatures was conducted of the friction, welding, and wear properties of various cage materials sliding against bearing materials of either the molybdenum tool-steel type or the standard SAE 52100 type. The latter material was used only for comparison purposes. In all cases, selection of the cage materials was based as much as possible on the three requirements other than compatibility previously listed. It should be emphasized that these studies provide fundamental information on the friction and wear properties of materials in bench tests. Additional studies are required in full-scale bearings both in test rigs and in actual turbine engines before final conclusions can be made with respect to use of these materials.

The apparatus used for these studies was essentially similar to that shown in figure 1 and described in reference 4. The studies for which results are presented in figures 9 to 13 were performed dry, that is, with no lubricant present, and the apparatus was modified so as to provide temperatures up to 1000° F by a suitable furnace. (Some of the data of fig. 12 were obtained with lubricant.)

Figure 9 shows the effect of temperature on friction and wear of a conventional (Alcop) bronze, an iron-silicon bronze (Mueller 803), and a cast pearlitic nodular iron. The bronze, which is a presently used cage material for aircraft turbine-engine bearings, shows a continuous increase in friction with higher temperatures; wear decreases with increasing temperatures to 450° F and then increases with higher temperatures. Experience has indicated that sliding velocities greater than that of these experiments would shift any critical region (such as the minimum wear point for the Alcop bronze) toward the origin on the temperature curve. Iron-silicon bronze showed no effect of temperature on friction and wear at temperatures below 600° F; at higher temperatures both friction and wear increased. The nodular iron had unstable friction coefficients at temperatures of 450° F and lower with spasmodic increases to values as high as unity; this friction behavior was

not accompanied by high wear. Friction of the nodular iron decreased slightly with increase in temperature, and above 600° F the friction was less than that for either of the bronzes. Wear of the nodular iron increased slightly with temperatures of 600° F and above, but the trend of increasing wear was considerably less than for the bronzes.

Previous data at high sliding velocities indicated that nickel alloys can have good resistance to wear and surface failure under conditions where surface oxidation can occur to form protective oxide films. It was suggested that this tendency might become more pronounced at high temperatures. Data for modified H monel, cast Inconel, and Nimonic 80 are presented in figure 10. These materials have physical properties that are not affected significantly by temperatures of 1000° F and higher. In general, these materials had high friction coefficients that decreased with increasing temperatures. The materials also showed decreased wear with increasing temperatures. The decrease in wear with increase in temperatures for H monel was quite gradual but for both the cast Inconel and the Nimonic 80 abrupt changes in wear rates were observed. As previously suggested, it is believed that the temperatures at which the low wear rates are obtained would be reduced by operation at high sliding velocities.

The importance of surface oxides to the functioning of nickel alloys was demonstrated by making a run with cast Inconel at 1000° F in an atmosphere where the availability of oxygen was reduced by displacing air with argon. The wear was increased by a factor of four, while only a slight increase in friction was observed. Also, as shown in figure 11, when cast Inconel which previously had been run for 1 hour at 1000° F to form an oxide film was run an additional hour at room temperature, the wear at room temperature (0.078×10^{-3} cu in.) was slightly less than one-tenth that obtained with a new specimen at room temperature (0.917×10^{-3} cu in.). Further, when an unused cast Inconel specimen was heated in molten caustic (NaOH) to obtain a preformed oxide film and was then run at room temperature, the wear (0.094×10^{-3} cu in.) was also approximately one-tenth that obtained under identical conditions (0.917×10^{-3} cu in.) with the untreated specimen of the same material.

The wear experienced with cast Inconel having an oxide film against M-10 steel at room temperature is less than one-fourth that obtained (0.447×10^{-3} cu in.) under the same conditions with the metal combination (Alcop bronze against SAE 52100 steel) currently being successfully used in bearings of full-scale engines. Wear for the various nickel alloys at 1000° F in air was approximately one-tenth that for iron-silicon bronze and was approximately one-half that of the iron-silicon bronze at 600° F. It is in the temperature range from 600° to 1000° F that nickel alloys merit particular consideration as cage materials.

It is suggested that it may be possible to obtain low wear both at low and at high temperatures by employing a nodular-type cast iron containing 20 percent nickel (Ni resist). The combined effect of the free graphite from the nodules and the possible surface film of nickel oxide could produce the desired result. An investigation of this material will be made in the near future.

The high friction of all the materials previously discussed is undesirable because it could cause excessive cage slip. Preliminary experiments conducted with a hot-pressed iron-silicon bronze containing MoS_2 were unsuccessful because of high wear resulting from poor physical properties; however, those experiments did show significantly reduced friction coefficients could be obtained. This approach appears promising and further research is in progress to obtain reduced wear as well as lower friction for this type of material.

Results of lubricated friction and wear runs at 450° F of various possible cage materials against M-10 tool steel obtained at a sliding velocity of 120 feet per minute with a load of 1200 grams are shown in figure 12. The lubricant was di(2-ethylhexyl) sebacate plus 0.5 percent phenothiazine (oxidation inhibitor). Data for these same materials run dry against M-10 tool steel at 450° F are also included for comparison purposes. The results show that, as expected, the lubricant served to decrease both friction and wear for all except the conventional bronze. The bulk temperature of the lubricant was so high that the specimens were believed to be operating in a range of incipient failure of boundary lubrication where the compatibility of materials is very important. The reduction in wear of the cast Inconel is particularly to be noted since its dry wear was rather high.

Since a number of promising tool steels have been considered for races and rolling elements of rolling contact bearings, data were obtained (fig. 13) showing friction and wear properties of cast Inconel sliding against these various possible bearing steels at both room temperature and 1000° F. Hardness of all steels was from Rockwell C-60 to C-63 at room temperature. The data of figure 13 did not indicate any important difference in the friction or wear of cast Inconel against SAE 52100 as compared to the various tool steels of equivalent hardness.

Cage and Bearing Designs

It has been shown (ref. 7) that considerable improvement in the operating characteristics and, in particular, in the limiting speeds of a bearing can be obtained by proper cage and bearing design. For example, in reference 7 it is shown that the combination of an outer-race-riding cage with a straight-through outer race (inner-race-guided rollers) gives the best over-all performance. The better performance of this design over both the conventional inner-race-riding cage type and the conventional outer-race-riding cage type with outer-race-guided rollers is a result of the relative ease of lubrication and cooling and of the adequate oil flow (exit) paths which minimize oil churning and friction losses. That is, the design principle emphasized in these results seems to be that of providing for easy flow of lubricant into, through, and out of the bearing. Under these conditions, both adequate lubrication and adequate cooling are obtained; this result tends to extend the limiting speed values. It is possible that application of this design principle will also assist in extending the high-temperature limitations.

In the studies of reference 8 it was shown that cage slip is extremely detrimental to bearing performance particularly under high-speed conditions. It was also shown that the use of a cage material which has a high friction coefficient might be particularly bad from the standpoint of inducing slip under extremely high speeds. These results would indicate that another extremely important property of a cage material is that of maintaining low friction coefficient under the expected conditions of operation. This point was not completely appreciated before the results of reference 8 were obtained, since it was felt that friction coefficient in itself was not a particularly important factor as long as the other performance characteristics of the cage material were satisfactory. It should be emphasized, however, that the friction coefficient is of primary importance only insofar as it influences slip within the bearing.

Temperature Ranges for Various Bearing Materials

A summary of the possible bearing materials (both for rolling contact and for cages) for operation over the four temperature ranges is given in table II. As shown, the present race and rolling element material (SAE 52100) and the present cage

material (silver-plated bronze) are suitable for temperature range 1. Likewise, the present bearing materials may be used in the interim until more information is available on the other materials for temperature range 2; this is, however, only an interim measure and the molybdenum tool steels and other cage materials (such as nodular iron and nickel alloys) may be used in the near future. For temperature ranges 3 and 4, the rolling contact bearings will undoubtedly employ tool steels for races and rolling elements and nickel alloys, and nodular iron or other special materials for the cages. Very little is known about the performance of these materials under these conditions; in consequence, much additional research is required (fatigue as well as friction and wear research) in temperature ranges 2, 3, and 4.

LUBRICATION METHODS

Bearings

Liquids. - The standard method employing liquid lubricants in a solid-jet lubrication system is well known. The limitations of this system are a function of both the stability of the liquid lubricants at high temperature and the bearing designs, as previously mentioned. Thus it may be possible to extend the high-temperature limitations slightly by use of liquids of better high-temperature stability or by improving the cage and bearing designs so as to permit free flow of lubricant through the bearing.

Solids. - Solids may be used for lubrication in essentially two manners: (a) by including the solid as a minor constituent of the material to be lubricated, and (b) by an air-mist system similar to that described in reference 9 or mixed with a volatile liquid carrier.

The principle of lubrication of materials at the interface of two rubbing surfaces by use of material within the structure of one of the two rubbing surfaces is well known. Many leaded materials such as the leaded bronzes are believed to operate in this fashion, and cast iron is a good example of this type of material since it is believed to be lubricated by graphite from within the structure. Similarly, materials can be made by the powder-metallurgy technique which employ, as a minor constituent, solid materials that are excellent lubricants. Results obtained on materials of this type (containing molybdenum disulfide, MoS_2 as the solid lubricant) are included in reference 10. As this reference shows, mixtures of silver, copper, and MoS_2 were quite effective in producing low friction and low wear under certain optimum concentrations of molybdenum disulfide. Under the conditions of the experiments of reference 10, this optimum concentration was approximately 7 to 10 percent. These results show that, even under extreme conditions of operating dry, it is possible to obtain moderate wear rates, complete absence of welding, and moderately low friction coefficients.

The results of reference 9 show that it is possible to obtain adequate lubrication of rolling contact bearings under either high temperatures (up to 1000°F) or at high speeds (up to DN values of 1,000,000). Unfortunately, it was not possible in the preliminary investigation of reference 9 to combine the two variables of high temperature and high speed, since the test rigs were limited in either case. The air-mist system of applying MoS_2 showed enough promise in this preliminary investigation to warrant additional research under the combined conditions of high speeds and high temperatures once a rig is obtained which is not limited in these respects.

Gases. - Because of the possible limitation imposed on engine life and/or engine design by the fatigue life of rolling contact bearings and of the limitations

imposed by inadequate high-temperature lubricants, hydrodynamic bearings employing air as the fluid have been considered as a possible solution to the high-temperature problem. Although the studies of reference 11 were limited to a nonrotating bearing, they did show nevertheless that the air bearing has some possibility for supporting load in a relatively stable manner at temperatures of 1000° F. Also, an air bearing has the prime advantage of showing an increase of load-carrying capacity as the temperature increases, in direct contrast to hydrodynamic bearings employing liquids which show a decrease in load-carrying capacity with an increase in temperature. It is believed that the air bearing may have some definite place in the field, in particular where the conditions of operation are so severe that rolling-contact bearings cannot be employed. Under these conditions, the advantages of the air bearing might outweigh its disadvantages of requiring small clearances, careful alignment, and a source of high-pressure air.

Gears

It is generally agreed that the load-carrying capacity of gears is increased by using lubricants of greater viscosity. As a consequence, for those gears such as turboprop engine reduction gears which are highly loaded and which are operating under marginal conditions of lubrication with present low-viscosity lubricants, it would appear that a change to higher-viscosity lubricants would be extremely desirable. This change can be performed in two ways: (1) by relaxing the low-temperature viscosity limits at present outlined in specification MIL-L-7808, and (2) by providing a lubricant which meets specification MIL-L-7808 low-temperature viscosity requirements but which has improved viscosity-temperature properties to such an extent that the viscosity at elevated temperature is appreciably higher than that of the present lubricants.

Present lubricants. - The present lubricants meeting specification MIL-L-7808 have load-carrying capacities on the Pratt & Whitney Ryder gear rig considerably less than the value which has been indicated to be that desired for effective gear lubrication in a typical turboprop engine. This desired value has been quoted as a load-carrying capacity equivalent to that of grade 1100 petroleum lubricant. In order to obtain a load-carrying capacity equivalent to grade 1100 with a diester, a high-viscosity fluid such as Esso laboratories E.E.L.-3A is required. The viscosity is such that the fluid is not pumpable at -65° F; in fact, it is impossible to measure viscosity at this temperature because the level of viscosity is so high. The quoted values of viscosity in centistokes for E.E.L.-3A are 7.6 at 210° F, 37.6 at 100° F, and 12,300 at -40° F. While E.E.L. - 3A has adequate load-carrying capacity, it cannot be pumped at -65° F; therefore, some other type of lubricant is necessary to meet both requirements.

Silicone-diester blends. - The NACA silicone-diester blends similar to those originally described in reference 4 may meet the two requirements of adequate load-carrying capacity and pumpability at low temperatures. One of these blends, SD-17, has a viscosity at -65° F of approximately 4000 centistokes and viscosity at 210° F approximately the same as that of grade 1100 oil as shown in table III. Table III presents data obtained through the excellent cooperation of Pratt & Whitney Aircraft and shows that the various lubricant properties other than load-carrying capacity are also promising. The various methods of evaluation are described in reference 12. Two points were noted by Pratt & Whitney: (1) high wear in the Shell four-ball wear machine, and (2) a high ash content. If the wear in the Shell four-ball wear machine is truly significant, it is believed this value can be reduced appreciably in the SD-17 blend by use of additives such as tricresyl phosphate. Data obtained by Fenske at the Pennsylvania State College (ref. 13, table 29) lead to the belief

that addition of relatively small quantities of tricresyl phosphate will markedly reduce wear as measured in the Shell four-ball wear machine.

A high ash content must be expected with synthetic materials that are basically inorganic in nature such as silicones. The important point to determine in this regard is whether or not this high ash content is actually detrimental to engine performance. Such studies are now in progress at the NACA.

Table IV presents IAE gear-test data obtained by the Esso European Laboratories through the very helpful cooperation of the Esso Laboratories Standard Oil Development Company of Linden, N. J. The data of table IV show that the load capacity of the NACA SD-17 silicone-diester blend compares very favorably with grade 1100 oil and the compounded diester PRL 3313.

CONCLUDING REMARKS

On the basis of the existing data, there appears little likelihood of obtaining liquids which can adequately lubricate bearings operating at temperatures of 1000° F. While this view is subject to modification based on the amount of cooling available for the lubricant, it is believed that it will be desirable in the design of future high-temperature engines to provide as little cooling for the lubricant as is practicable. Some of the data show that considerable gain (up to 200° F) in high-temperature lubrication by liquids can be made by relaxing the low-temperature specification, that is, by use of lubricants of higher viscosity. The use of solids and gases as lubricants at temperatures of the order of 1000° F appears to show some promise. Considerable research must be performed to prove both of these lubricant types.

On the basis of the data available, it appears that promising materials exist for use as rolling elements and races of rolling contact bearings. These materials, however, require more complete evaluation, particularly with respect to fatigue.

Rolling-contact bearing cage materials are already a problem in present engines; this problem is expected to become worse as temperatures increase. Some cage materials appear promising; here again, considerable research and evaluation is necessary to establish the surface compatibility of these materials with the bearing race materials.

Some gain in the performance characteristics and possible extensions in the limitations of existing rolling contact bearings can be obtained by cage designs which are based on the principle of providing an easy flow path of lubricant into and through the bearing. The use of silicone-diester blends as gear lubricants may be of decided advantage because the silicone-diester blends meet the dual requirements of adequate pumpability at low temperatures and good load-carrying capacity in gears.

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TABLE I. - PROPERTIES OF EXPERIMENTAL FLUIDS

Fluid	Viscosity, centistokes, at -				A.S.T.M. pour point, °F	C.O.C. flash point, °F	C.O.C. fire point, °F
	-65° F	-40° F	100° F	210° F			
Petroleum: MIL-O-6081a (grade 1010) ^a	40,000	-----	9.95	2.47	<-70	300	---
Diesters:							
Di(2-ethylhexyl) sebacate ^a	8,297	-----	12.78	3.32	<-70	440	472
Di(2-ethylhexyl) sebacate plus additives ^{b,c,d,e}	16,000	2,700	20.8	5.3	<-75	450	475
Polyalkylene glycols (designated by viscosity at 100° F):							
Water-soluble							
55 SUS (8.9 centistokes) ^e	-----	1,800	8.9	2.4	-85	260	285
190 SUS (41 centistokes) ^e	-----	30,000	41	8.2	-45	410	480
660 SUS (143 centistokes) ^e	-----	26,000	143	26.2	-30	430	545
3520 SUS (762 centistokes) ^e	-----	762		120	-20	440	545
Water-insoluble							
50 SUS (7.4 centistokes) ^{e,g}	4587	905	8	2.52	<-70	270	335
Phosphonate: Dioctyl isooctene phosphonate ^a	-----	ⁱ 9,003	12.22	2.77	-90	---	---
Silicate: Tetra (2-ethylhexyl) silicate	1,400	260	6.8	2.4	<-100	395	450
Silicone-diester blend, SD-17							
$\frac{1}{3}$ di(2-ethylhexyl) sebacate ^{a,h} plus $\frac{2}{3}$ methylphenyl polysiloxane (100 centistokes at 77° F)	3,750	1,050	42	14.2	<-80	450	500

^aMeasured values.^b4 percent methacrylate polymer.^c5 percent tricresyl phosphate.^d0.5 percent phenothiazine.^eManufacturer's data.^fAt -20° F.^gContains oxidation inhibitor.^hParts by volume.ⁱAt -50° F.

TABLE II. - TYPICAL LUBRICANTS AND BEARING MATERIALS FOR VARIOUS TEMPERATURE RANGES

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Temperature range	1	2	3	4
Maximum operating temperature, °F				
Bearing	< 350	500	750	1000
Bulk lubricant	250	300	400	----
Minimum temperature, °F	-65	-65	-65	-65
Possible lubricants	Liquid-petroleums	Liquid-diesters and other synthetics	Solids or gases	
			Liquids in open or closed systems	
Research required on lubricants	None	High temperature stability and load-carrying capacity (for turboprops)	High temperature stability and lubrication in open or closed systems	
Possible rolling contact bearing materials	SAE 52100	Tool steels		
For races and rolling elements		SAE 52100 (interim material)		
For cages	Silver-plated bronze	Nickel alloys		
		Nodular iron		Powdered metals with solid lubricants
		Silver-plated bronze (interim material)		
Research required on bearing materials	None	Fatigue research		
For races and rolling element materials				
For cages	None	Friction and wear research		

NACA RM E54D27

TABLE III. - RESULTS OF PRELIMINARY PRATT & WHITNEY TESTS ON NACA SD-17 LUBRICANT

	Results of NACA Oil SD-17 ^a	Specification requirements PWA 521A
Lubrication		
Viscosity, centistokes, at:		
-65° F	4242	13,000 max.
-40° F	1175	3000 max.
100° F	42.59	11 min.
210° F	14.29	3 min.
Viscosity index	171	--
Pour point, °F	-85	-75 max.
Gear scuffing, lb/in.	3700, 2675	1700 min.
Shell four-ball:		
Min. load for immediate seizing, kg	40	--
Weld point, kg	110	130 min.
Corrosion		
Sulfur, percent	0.05	--
Neutralization number	0.00	--
Reaction	Neutral	--
Copper-strip corrosion	Passed	No pitting
Corrosion-oxidation stability		No pitting; slight stain permitted
Corrosion, weight change, mg/cm ²		±0.2 max.
Copper - stained	0.10	
Steel - stained	0.06	
Aluminum - passed	0.02	
Magnesium - passed	0.09	
Silver plate - passed	0.05	
Viscosity at 100° F, centistokes	43.28	--
Change from original, percent	+1.6	-5 to +15
Neutralization number	1.5	--
Change from original	1.5	1.0 max.
Average lead weight loss, (MROC), g	0.0070	0.30 max.
Neutralization number (after MROC)	0.10	--
Corrosion:		
Mallory 100 (wt. loss, mg)	8, 7	10 max.
Silver (wt. loss, mg)	5, 3	10 max.
Deposition		
Precipitation number	0.05	--
Carbon residue, percent	2.29	--
Ash, mg/20 g	4597 (23 percent)	--
Additives, metals (spectroscopic)	Heavy Si; Low Cu, Ag	Corrosion inhibitors and pour-point depressants permitted
Panel coking (wt. inc., mg)	25	100 max.
Miscellaneous		
Gravity, °API, 60° F	13.9	--
Gravity, specific, 60/60° F	0.9732	--
Saponification number	74.3	--
Flash point, °F	455	365 min.
Fire point, °F	505	--
Low-temperature stability (72 hr at -65° F)	Passed	No separation, or gelling
Evaporation loss at 275° F, percent by weight	0.515	10 max.

^aContains PTZ (0.5 percent) only.

TABLE IV. - RESULTS OF IAE GEAR TESTS ON SPECIAL LUBRICANTS

(PERFORMED FOR NACA BY ESSO EUROPEAN LABORATORIES THROUGH
COOPERATION OF ESSO LABORATORIES, LINDEN, N. J.)

[2000 rpm: 1/2 pint/min: 90° C: B.S.En. 39 gears.]

Lubricant -	Failure load as percent of reference oil
Esso Aviation Oil 100 (grade 1100, petroleum, ref. oil)	100
Compounded diester (PRL 3313)	170
Silicone-diester blend (NACA SD-17)	230 (no scuffing)

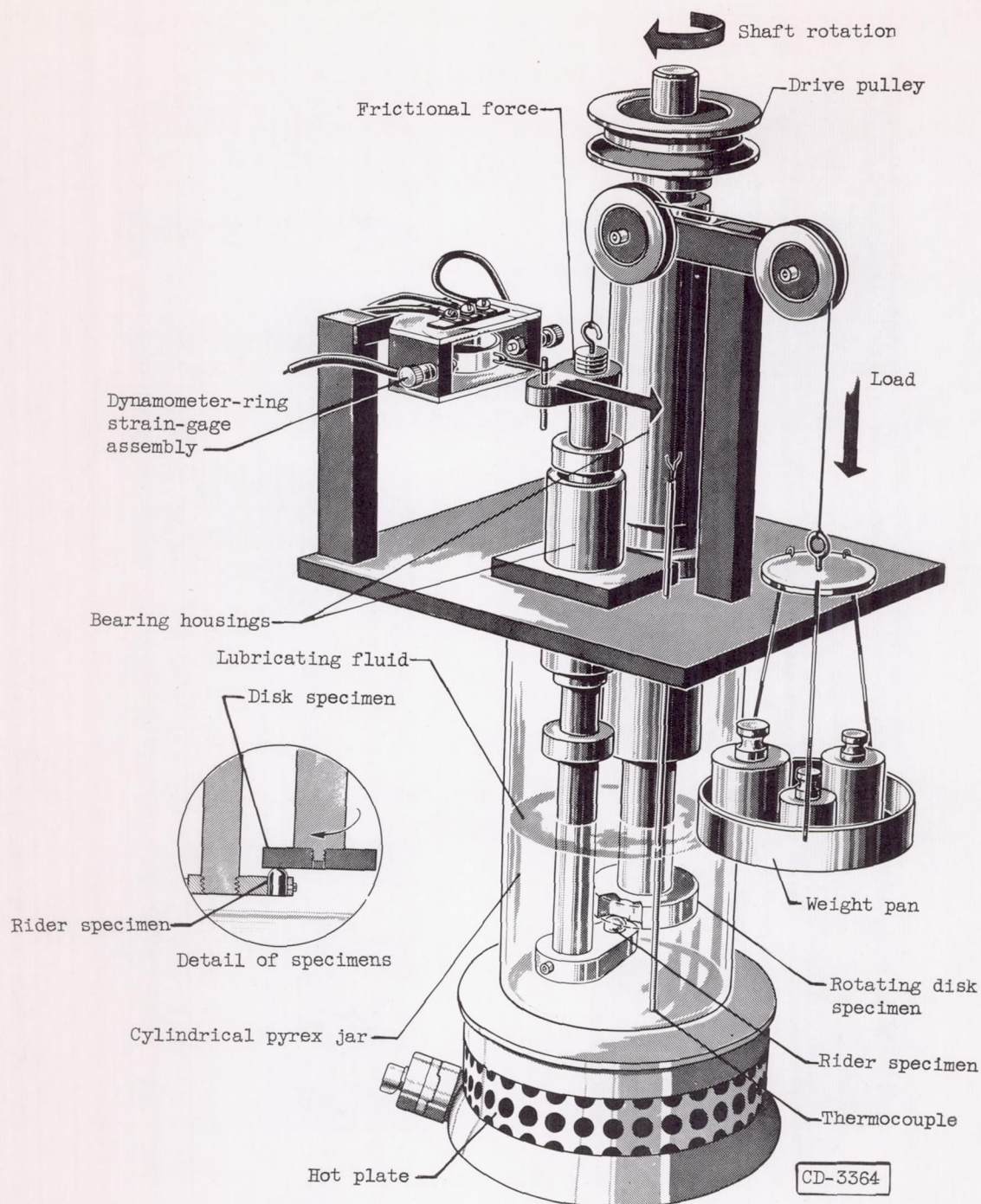
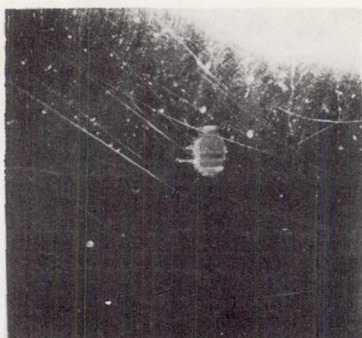


Figure 1. - Schematic diagram of friction apparatus for studying boundary lubrication by bulk lubricants.

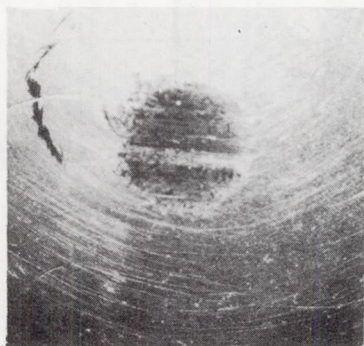
Rider specimen



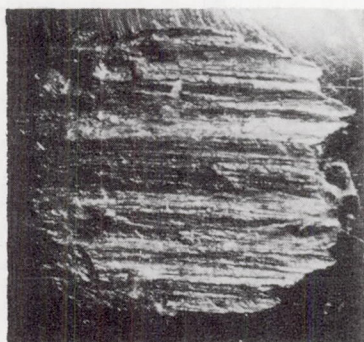
Disk specimen



(a) Effective lubrication



(b) Incipient failure



(c) Mass failure

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Figure 2. - Photomicrographs showing typical wear areas on rider and disk specimens. X15.

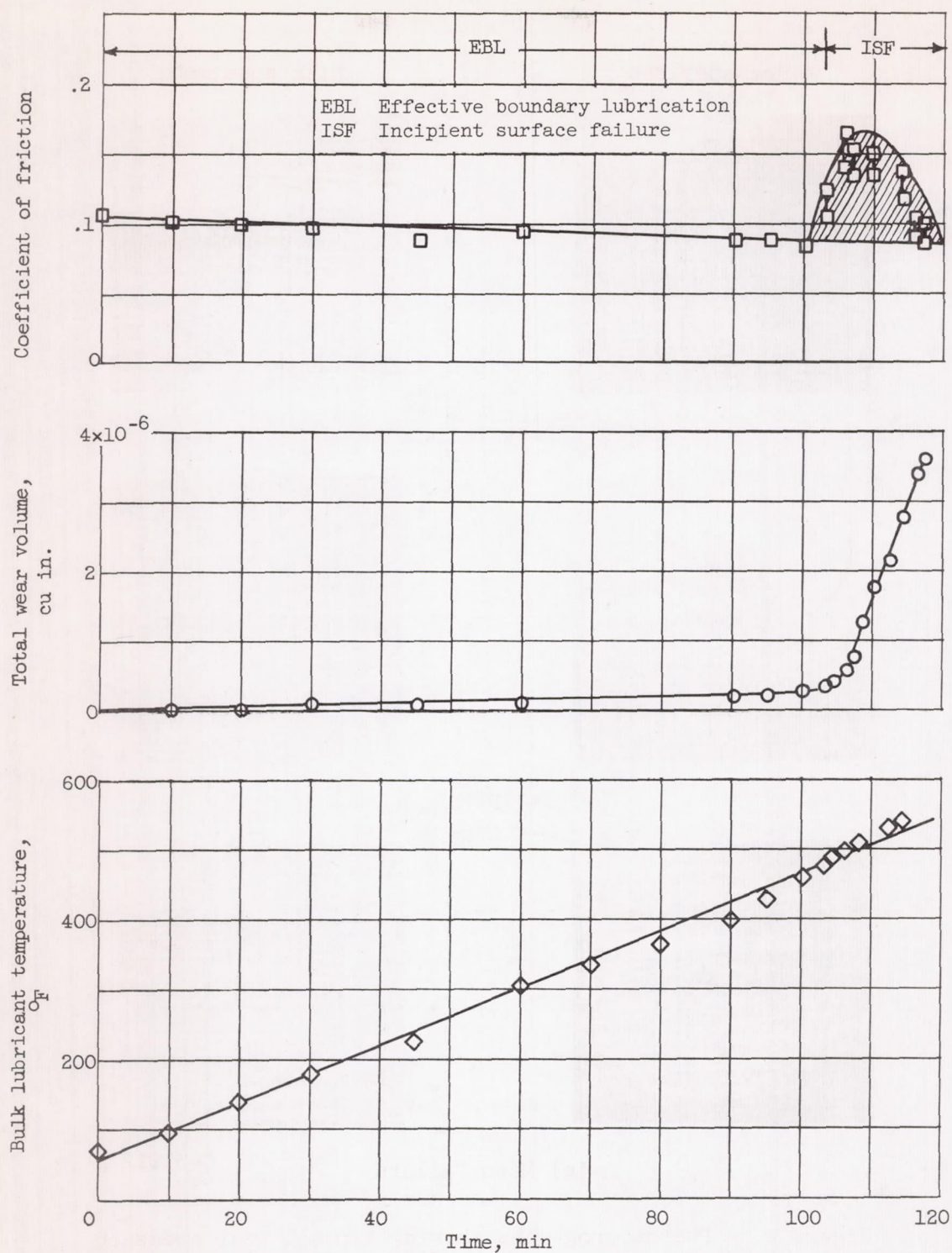
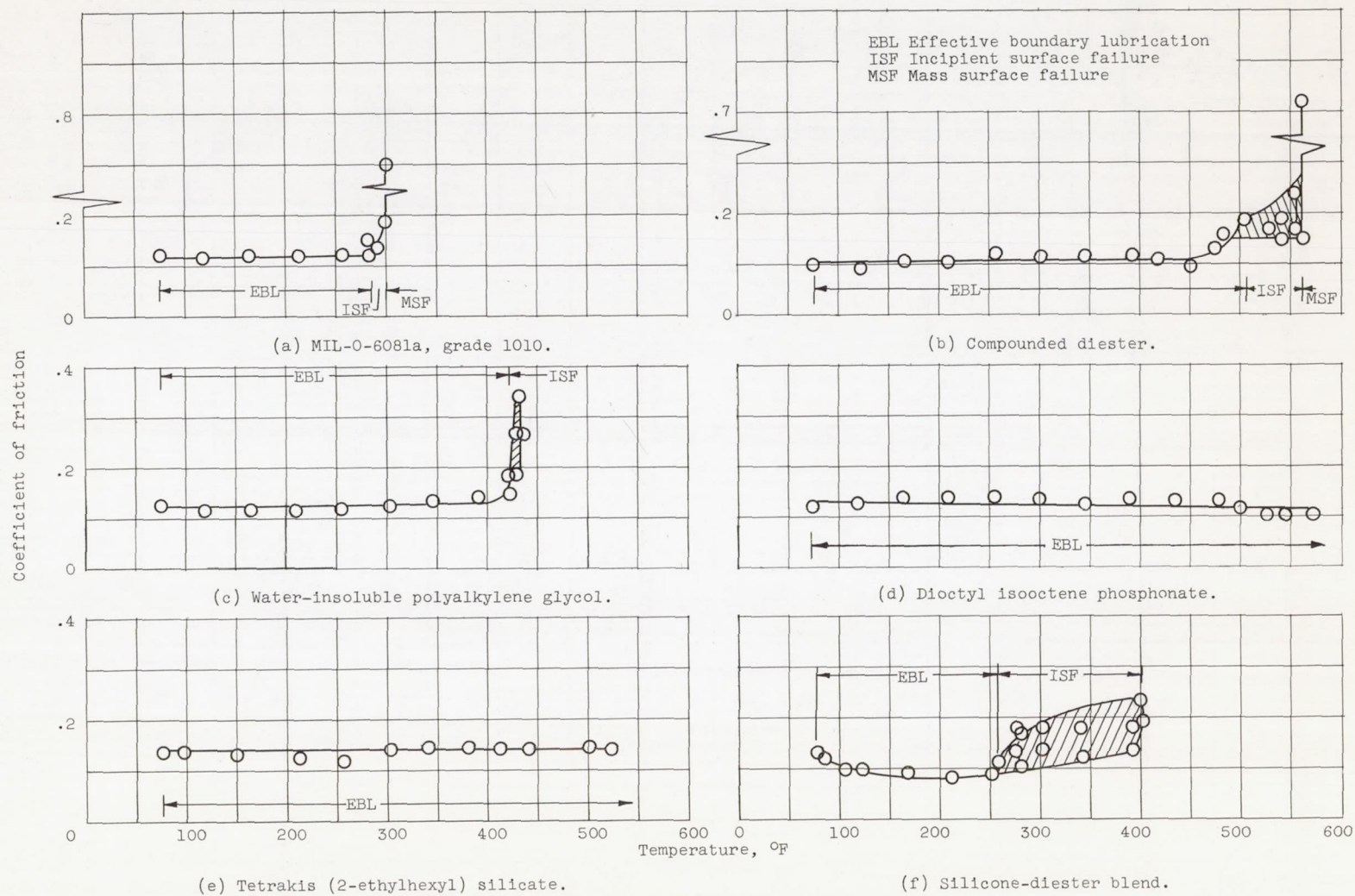


Figure 3. - Relation between unstable friction (cross-hatched area) and wear for typical diester lubricant at increasing bulk temperatures. Load, 1000 grams; sliding velocity, 120 feet per minute.



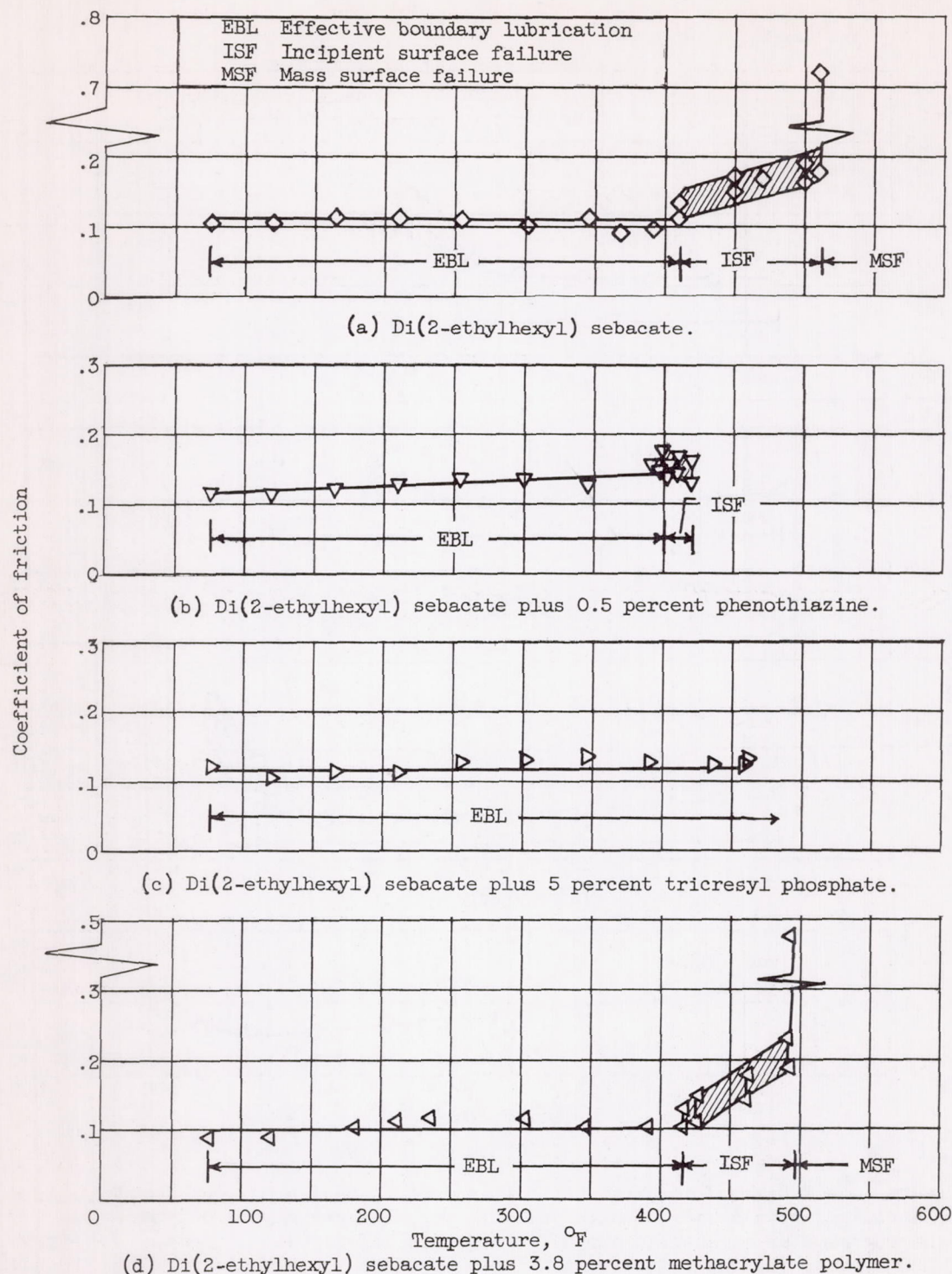


Figure 5. - Effect of temperature on friction of steel specimens boundary-lubricated with di(2-ethylhexyl) sebacate containing various additives. Load, 1000 grams; sliding velocity, 120 feet per minute.

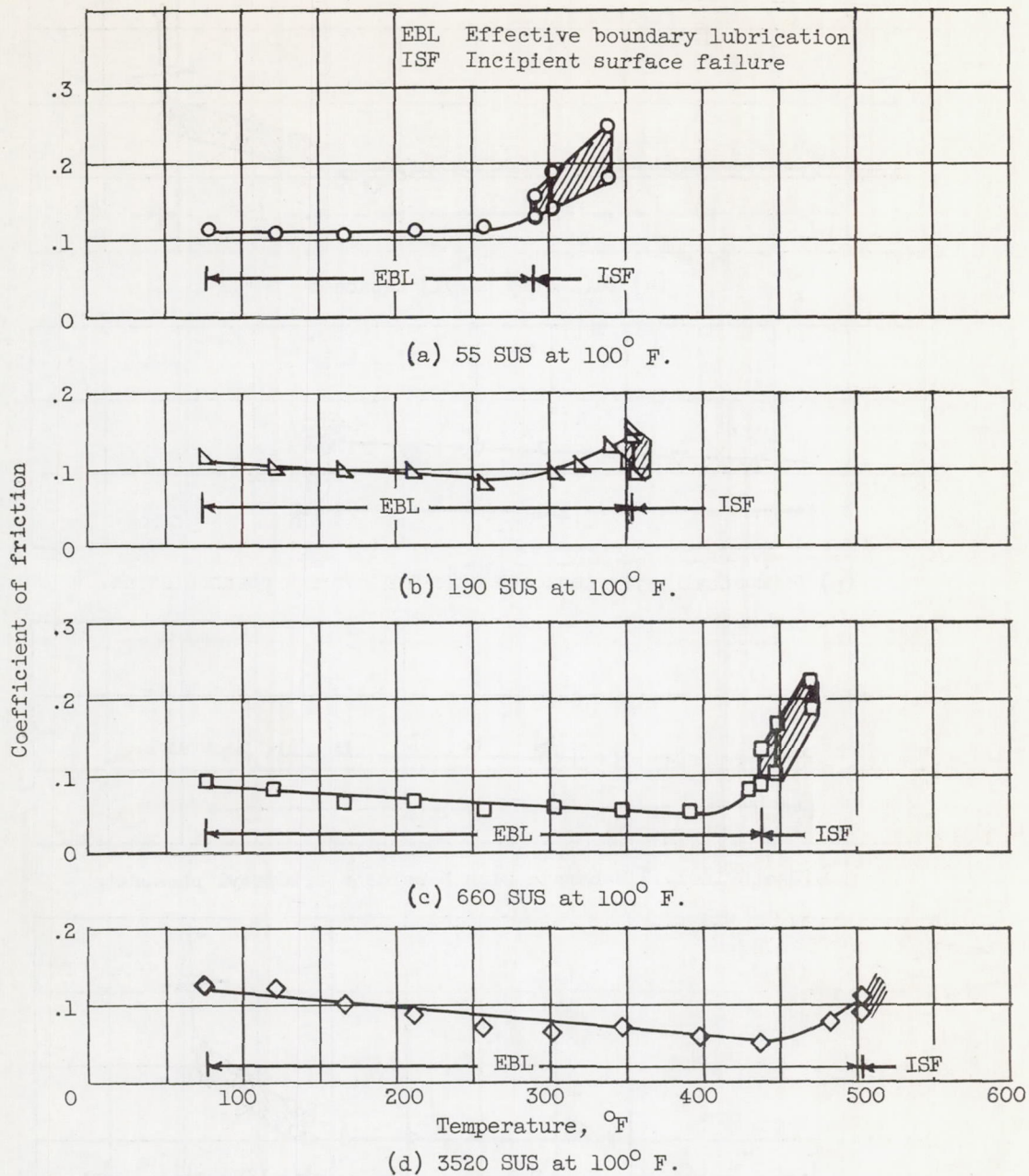
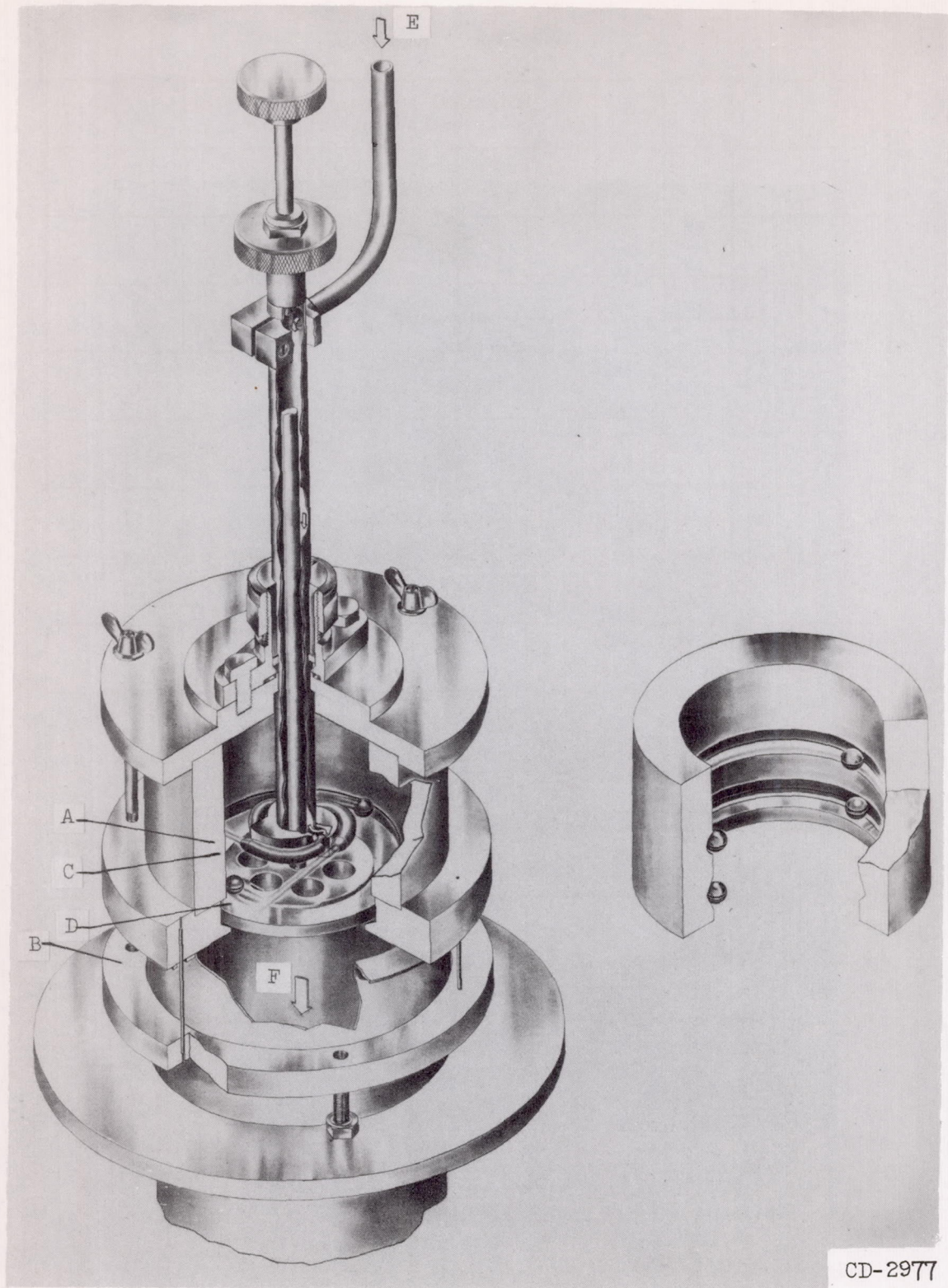


Figure 6. - Effect of temperature on friction of steel specimens boundary-lubricated with series of water-soluble polyalkylene glycol polymers having similar structures and differing only in viscosity. Load, 1000 grams; sliding velocity, 120 feet per minute.



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Figure 7. - The fatigue spin rig.

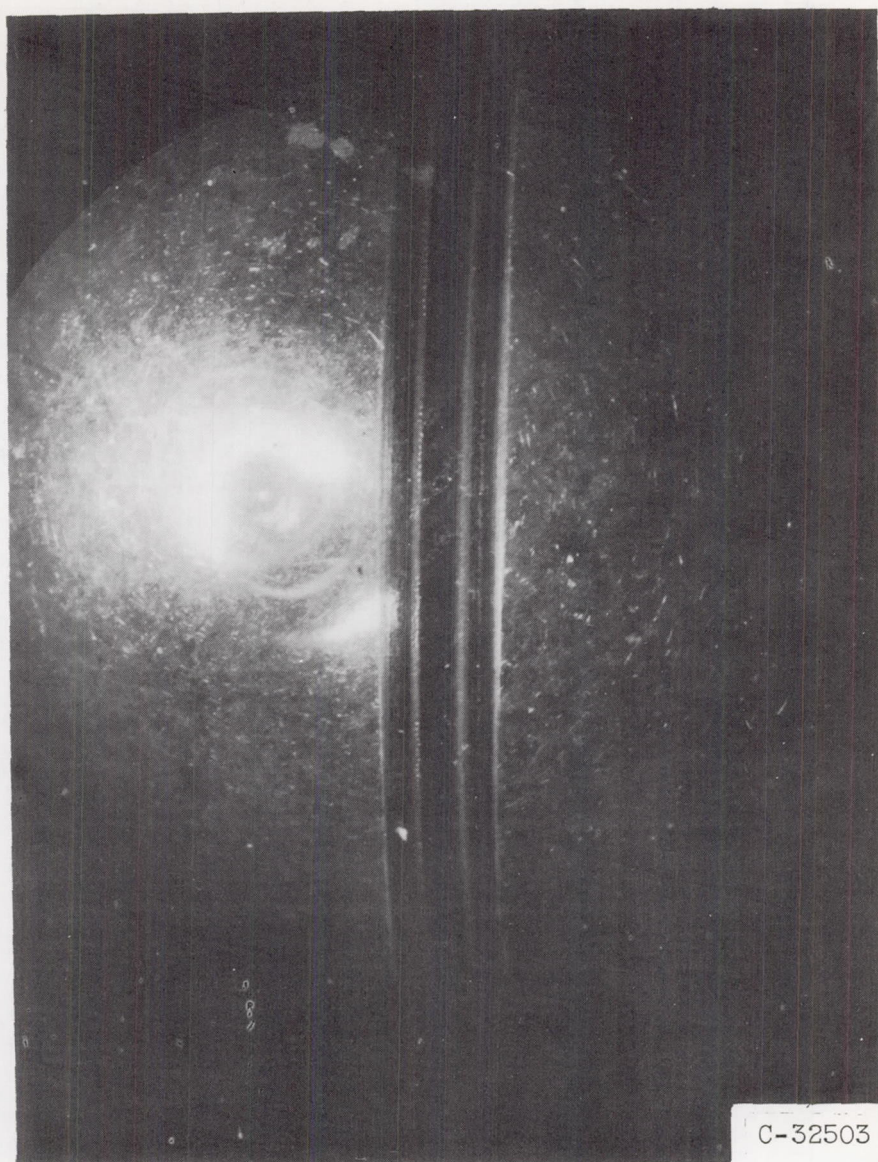


Figure 8. - Representative ball path obtained in fatigue spin rig.
Ball cycles, 1.358×10^6 ; maximum compressive surface pressure
(Hertz), 735,000 psi.

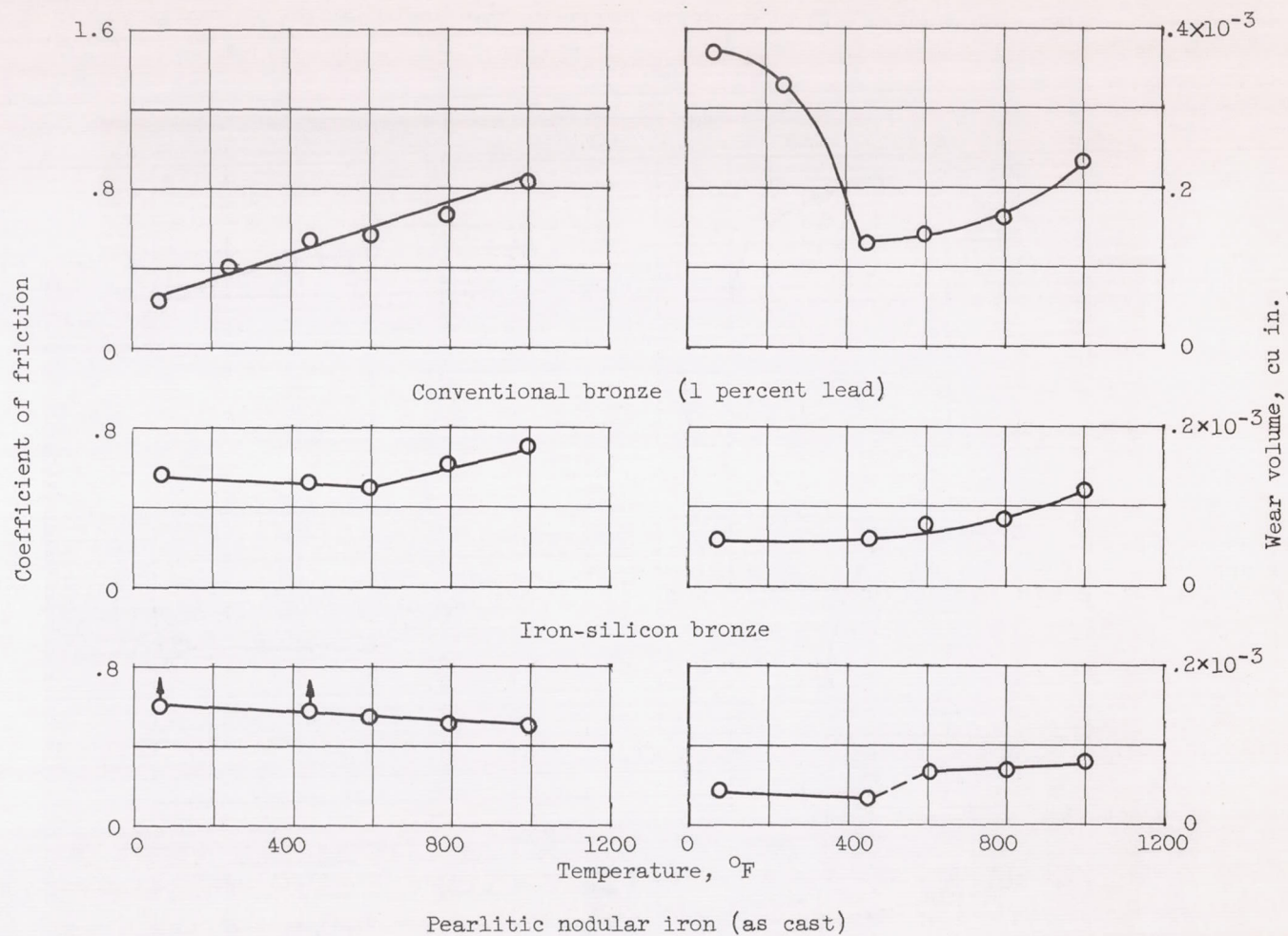


Figure 9. - Friction and wear of various materials at high temperature without lubrication.

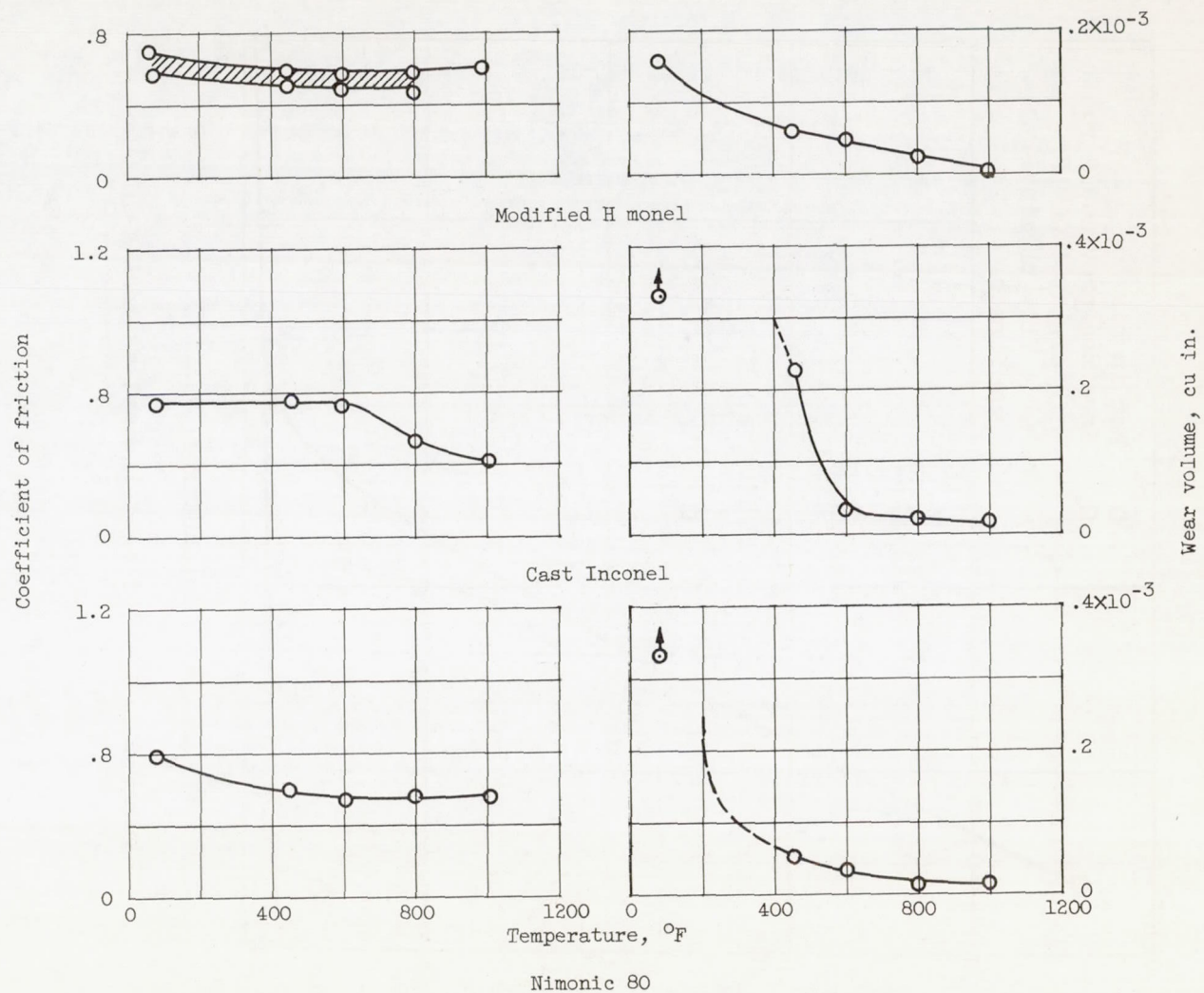


Figure 10. - Friction and wear of various materials at high temperature without lubrication.

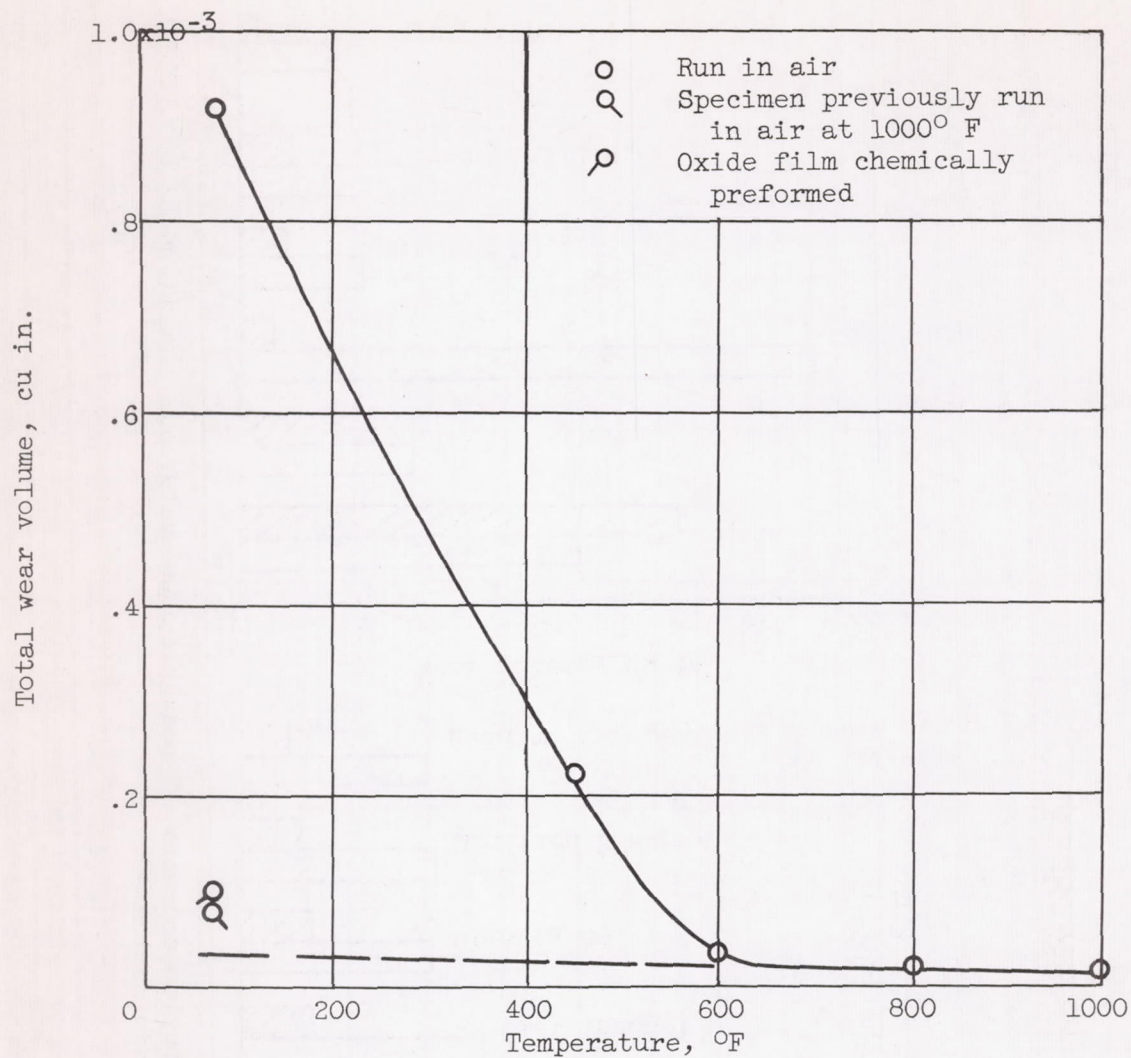
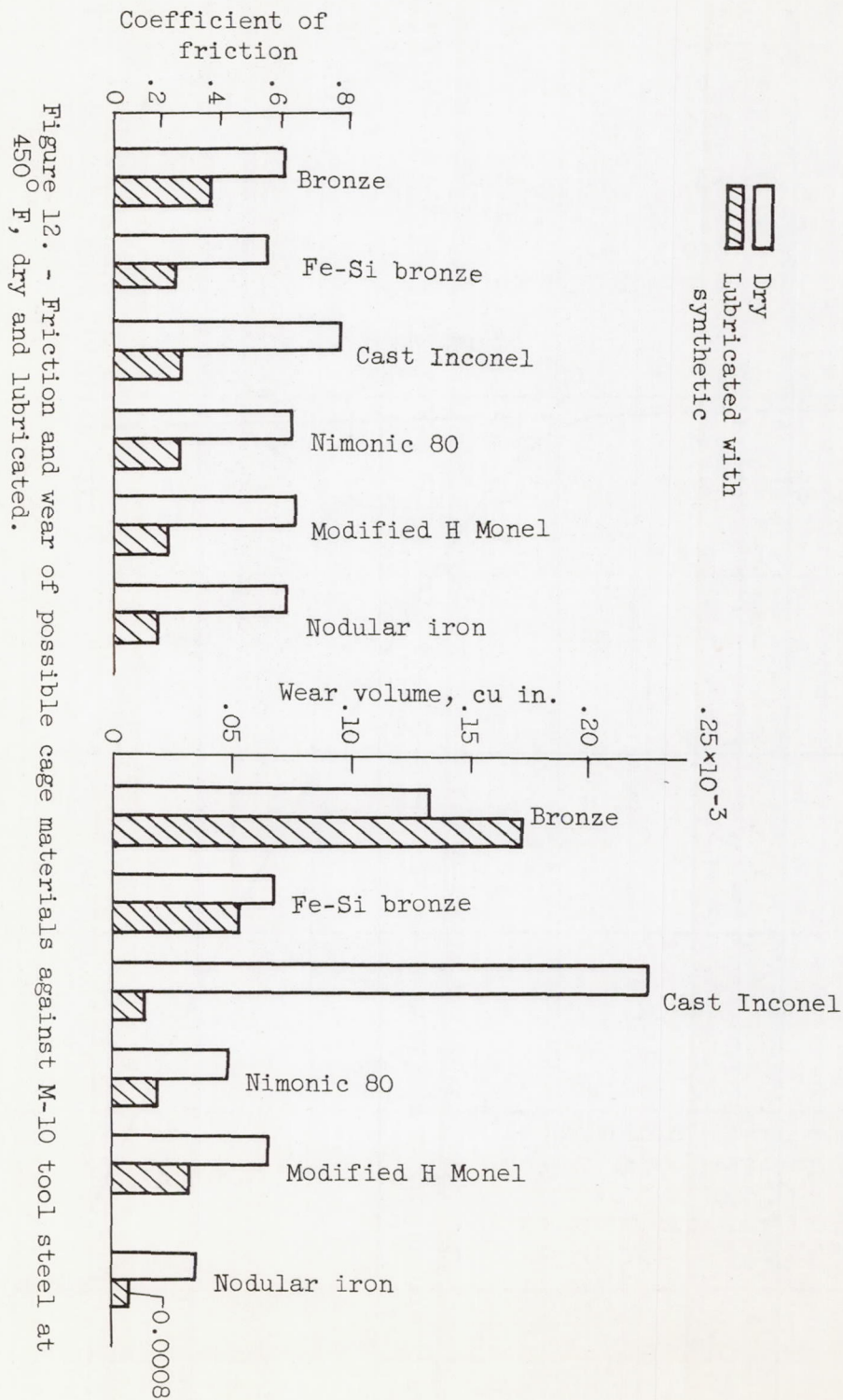


Figure 11. - Wear of cast inconel against M-10, dry.



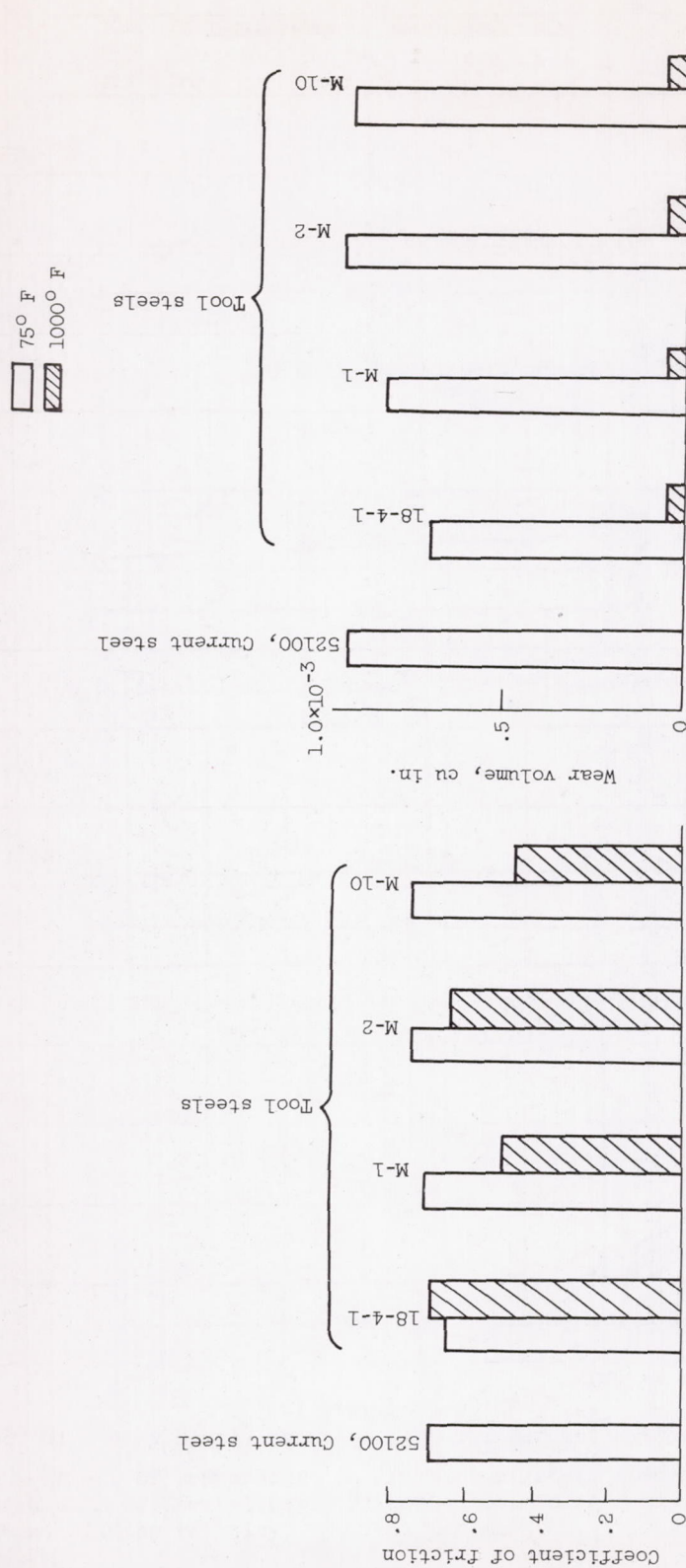


Figure 13. - Friction and wear of possible race and rolling element materials against Inconel, dry.

APPENDIX G

HIGH-TEMPERATURE BEARING PROBLEMS

By F. W. Wellons

In establishing material specifications for main or accessory case bearings in the hot locations of new high-performance engines, it is first necessary to examine the effect of time and temperature on the physical and metallurgical characteristics of conventional SAE 52100 bearing steel. A through-hardened SAE 52100 bearing ring which has been oil-quenched from an austenitizing temperature of approximately 840°C will contain 4 to 7 percent residual austenite finely emulsified throughout the martensitic structure. The rate of transformation for this residual austenite to martensite can be increased by either tempering at elevated temperature or by deep freezing in the -100°F range. Superimposed on the austenitic transformation, which is accompanied by a volumetric increase, there is a martensitic change of state from a tetragonal to cubic structure that is accompanied by a decrease in specific volume.

The response to temperature and time for a quenched SAE 52100 ring is schematically demonstrated by the several curves on figure 1. A low-temperature draw at 175°C is sufficient for the martensitic state change, but an extremely long time would be required to complete the breakdown of residual austenite. If the tempering temperature is increased to 200°C , the martensitic change is much more rapid and superimposed on the austenitic transformation. In this case the total shrinkage is not so great as for the 175°C draw. At 240°C the initial shrinkage is almost instantaneous followed by a rapid growth reaching a maximum in approximately four hours. An additional curve is shown to indicate the low-temperature austenitic transformation which results in a very hard, completely martensitic structure that would be essentially unstable even at room temperature. If the cold-treated bearing ring was subsequently subjected to running temperatures in the range of 175°C , the martensitic state change would be almost immediate, causing a reduction in volume, that is, a shrinkage of the bearing parts; consequently, a subsequent low-temperature draw after the deep-freeze treatment, as indicated by the dotted line, is necessary for complete dimensional stability. Long-time tempering tests at 240°C on parts given the stabilizing treatments described by figure 1 have revealed a decrease in specific volume which is generally attributed to precipitation of carbides towards a globular structure. However, this effect requires considerable time and is of no practical concern unless a bearing initially stabilized at 250°C (482°F) is intended for long-time service at temperatures above 400°F .

A typical tempering curve for a series of SAE 52100 specimens oil-quenched from 843°C is shown in figure 2. Each specimen was held at the indicated draw temperature for 1 hour and the hardness measured after cooling to room temperature. Comparing the curves of figures 1 and 2 shows that the draw temperature must take into consideration both the required degree of dimensional stability and the minimum acceptable ring hardness. Though a cold treatment followed by a subsequent low-temperature draw will give dimensional stability and at the same time a higher ring hardness than obtained through a single high-temperature draw, the metallurgical transformation is so drastic that there is considerable loss from cracked bearing rings both during the quench and later when the bearings are in service. Conversely, rings stabilized by a 250°C draw will be softer than the cold-treated rings and consequently more susceptible to ball and roller denting. Comparative endurance tests on several lots of bearings, which were identical in all respects except that

one-half had been stabilized through a cold treatment and the other half had been stabilized by a high-temperature draw for a minimum hardness of Rockwell C-58, did not show any difference in endurance life.

A stabilizing heat treatment for SAE 52100, which specifies a 4-hour draw at 235° to 240° C, resulted in a hardness of Rockwell C-58 to C-61. Although this treatment assures adequate dimensional stability for operating temperatures up to 350° F, recent experience with high soakback temperatures after engine shutdown demonstrated that 4 hours was not sufficient for complete stabilization. An 8-hour cycle at 250° C for SKF No. 1 steel or 240° C for SAE 52100 is desirable for the complete dimensional stabilization of bearings which are intended for operation where the temperatures may reach 400° F or higher.

Heretofore, practically all concern for temperature effects on bearing geometry has been centered on the turbine main shaft bearings, but in some new engines essentially standard ball and roller bearings supporting the accessory drives are either located in hot regions of the engine or are lubricated by hot scavenge oil from the main bearings. Normally these smaller bearings do not receive a stabilizing heat treatment because of their susceptibility to denting during manufacturing and in service. Test groups of typical accessory case bearings as shown in figure 3 were accurately checked for dimensional change after various periods at 500° F, and the average diameter changes for the bearing rings as computed from average diameters of the bore and outside diameter are plotted in figures 4(a) to (e). The radial looseness for each size radial bearing was checked by gage under a ± 5 -kilogram measuring load after each tempering interval as shown on figure 4(f). Measurements of less than 5 microns (0.0002 in.) are probably due to yield in the parts and do not reflect actual internal clearance. Figure 4(b), covering a 6910J ball bearing is particularly interesting because the outer ring in this case does not receive a stabilizing heat treatment while the inner ring (50-mm I.D.) is regularly stabilized. Hardness of the bearings of figure 3 is shown in figures 4(g) and (h).

Although low-temperature conditions are unusual for either the main or gear case bearings in aircraft turbines, some bearings in the airplane or engine accessories which are used intermittently in flight might be subject to outside air temperatures. Subzero tests with standard nonstabilized bearings did not show any appreciable dimensional change after 1 hour at either -40° C or -60° C but after 24 hours at -72° C the maximum growth amounted to approximately 0.001 inch per inch.

In the interests of economy, a program was recently initiated to determine if main shaft roller bearings currently made from high-temperature steel for operation at 500° F could be replaced by suitably heat-treated bearings of SAE 52100. A group of outer races made from SAE 52100 with a nominal outside diameter of 120 millimeters (4.7244 in.) were numbered consecutively, then carefully measured on the outside diameter and subjected to a tempering temperature of 600° F in groups of four for 1, 2, 4, 8 and 16 hours, respectively. After this experimental tempering treatment, all groups were heated to 500° F for 1, 2, 4, 8 and 16 hours with an outside diameter dimensional check after each cycle. Consequently, all rings were subjected to a total of 31 hours at 500° F. The dimensional changes and final hardness produced by the 500° F soak were as follows:

Group no.	Tempering time at 600° F, hr	Av. Rockwell-C hardness	Outside diameter change with time at 500° F in 0.001 mm (0.00004")					Av. Rockwell-C hardness
			1 hr	2 hr	4 hr	8 hr	16 hr	
1	1	56.1	-0.1	-0.3	-0.6	-0.7	-1.4	56.1
2	2	56.0	0	+0.1	+0.4	+0.1	+0.3	56.0
3	4	55.2	-0.1	0	+0.1	0	+0.1	55.2
4	8	55.0	+0.1	-0.2	-0.2	+0.4	+0.3	55.0
5	16	55.0	+0.3	-0.4	+0.4	+0.4	+0.4	55.0

From this table it will be noted that rings tempered at 600° F for times varying from 2 to 16 hours were quite stable after 31 hours at 500° F. As tempering times in excess of 2 hours at 600° F appear to have no influence on the dimensional stability at 500° F, 2 hours at 600° F, which appeared to have a slight hardness advantage, was selected for the sample roller bearings furnished for actual engine testing.

At the meeting of the Subcommittee on Lubrication and Wear in March 1953, a summary was distributed describing studies of high-temperature materials for turbine main shaft bearings; the hot hardness curves (figs. 5 and 6) have been prepared to supplement that information. The No. 3 steel is the same as SAE 52100, while 1 and 2 are of similar analysis with slight modifications for improved deep-hardening characteristics. The M series steels are well known in the high-temperature field and are covered in detail through the data presented at the last subcommittee meeting. The last four steels listed on figure 5 are proprietary analyses that either have been considered or have actually been used for high-temperature applications in current engines. Figure 6 was prepared to demonstrate the difference in hardness for the three analyses normally used for turbine main bearings in the as-quenched state and also after receiving a stabilizing heat treatment. For the stabilized rings the hot hardness is essentially unaffected at temperatures up to the tempering range, after which it falls off at the same rate as for the quenched but nontempered ring.

One of the most promising developments for improving ball and roller bearing reliability through better material cleanliness has been the erection of several furnaces for melting and pouring metals in a vacuum. In this process the desired analysis can either be built up in the furnace similarly to electric furnace practice or stock of the desired analysis can be remelted. Preliminary tests with ball bearings made from vacuum-melted SAE 52100 have been completed and further tests are being conducted to determine whether the endurance life is superior to that of conventional SAE 52100. The average life for a test lot of 6309 (45-mm-bore deep-groove ball bearing inner races made from vacuum-melted steel) was rated average to poor when compared to similar results for rings made from commercial SAE 52100. The life for 1309 (100-mm-outside-diameter self-aligning ball bearing outer races made from vacuum-melted SAE 52100) was comparable to results obtained in the laboratory with the best heats of conventionally melted bearing steels. However, the most impressive results have been with several bearing sets of 0.5-inch-diameter vacuum-melted steel balls which are still running after each ball set has failed one or more race sets. Normally, a set of balls previously run in a bearing where the rings have spalled will fail before flaking of the second ring set. Ball failures in such cases are aggravated by fragment denting from the prior raceway spalling, so that the survival of the vacuum-melted steel balls after several such race flakings is quite encouraging.

A second series of tests with 30 rings of each test size made from a different vacuum-melted SAE 52100 ingot is now underway. Although only a few bearings from this second lot of material have been run to failure, it would appear that the endurance pattern will be quite similar to that for the initial test group.

Because of restricted furnace capacity, the supply of vacuum-melted steel is quite limited, and the ingot cost is approximately 15 to 20 times that for SAE 52100. Consequently, the vacuum-melting process offers a particularly attractive source for high-temperature steels in those applications that are beyond the capabilities of SAE 52100. Generally, the demand for high-temperature bearings does not justify obtaining full heats, so that bar stock for ring forgings must be obtained through jobbing sources and consequently is of only average cleanliness. Inasmuch as tool steel costs are generally five to seven times that for normal bearing materials, the cost penalty for the vacuum melting and the limited ingot size (500 lb maximum at present), are not so objectionable as in the case of SAE 52100.

A program has been established in the SKF laboratory under contract with the Wright Air Development Center to obtain endurance data for the high-speed, high-temperature main thrust bearings in aircraft gas turbines. This program, which is already under way, is organized along three phases. First, a conventional test lot of thirty 6309 ball bearings made with rings and balls of M10 high-temperature steel will be endurance-tested on existing laboratory equipment under standardized test conditions. The results of this program will give a comparative picture of M10 and SAE 52100 for ordinary temperature, speeds, and loads. As soon as these tests are completed, the test rigs will be modified to run an identical lot of M10 6309 bearings under the same speeds and loads but at the highest possible temperature consistent with available lubricants.

Under this same program, one high-speed test rig capable of running 100-millimeter-bore ball bearings under pure thrust at speeds up to 15,000 rpm has been assembled and several trial runs made with non-test bearings. Most parts for the other two machines covered by the contract have been ordered, and the battery of three machines will be first used for the high-speed endurance testing of basic 7020 size (100-mm bore) precision angular contact bearings which are the same style as used for the main thrust bearing in aircraft turbines. The rings, races, and balls for this first lot of 30 high-speed test bearings will be made from SAE 52100 steel and the test conditions will be controlled by a circulating oil system to limit the maximum operating temperature to 350° F. This phase is primarily concerned with the effect of bearing geometry on endurance life at very high speed.

The final phase of this program will be the high-speed testing of angular contact ball bearings following the optimum geometry as developed during phase 2, but with rings and balls made from high-temperature steel. These tests will be run at approximately 15,000 rpm and cartridge heaters will be used to maintain ambient temperatures up to 750° F depending on available lubricants.

The characteristic aircraft power plant bearing application is an optimistic compromise of minimum bearing size with maximum load, speed, and temperature. Figure 7(a) shows a very interesting mounting using a double row, self-aligning roller bearing inner-race assembly for a pinion bearing, with the outer-race sphere ground integral with the high-speed pinion bore. Both 75- and 90-millimeter-inside-diameter pinion bearings of this type have been successfully operated in a fixed-cage planetary with a maximum DN value of 675,000. These pinion bearings use essentially standard inner-race, cage, and roller assemblies modified for high-speed operation with radial lubrication holes drilled through the center flange for positive lubrication of the cage support surfaces and the roller guide flanges.

A typical tandem mounting of two split inner-race ball bearings for the main thrust location in an advanced aircraft turbine is shown in figure 7(b). To insure equal load distribution, very close manufacturing controls are necessary to maintain the same contact angle for each bearing and to equalize their yield under thrust. As one bearing will tend to pump and the other to repel, a stationary baffle is inserted between the two outer rings with separate metering jets for lubricating each bearing. Relatively high oil flows are required to maintain a safe bearing temperature so that ample scavenging drains are necessary to avoid excessive heat from oil churning.

A high-precision angular contact bearing developed for an extremely high-speed, though not necessarily high-temperature, application is shown in figure 7(c). Laminated phenolic resins such as bakelite, micarta, and synthane, have been very successful for cages of high-speed antifriction bearings primarily because of their light weight and inherent self-lubricating properties. However, these materials have very low tensile strength, and preliminary high-speed testing with conventional precision angular contact bearings resulted in cage failures across the ball pocket, which were attributed to centrifugal strain. Consequently, an experimental cage was developed by shrinking a thin brass sleeve around a suitable nonmetallic cage blank and riveting this ring in place at two diametrically opposite locations. Previous studies had shown that the phenolic materials will distort with temperature as the volatile elements are driven off, so that some positive method, such as riveting, was necessary in addition to the interference shrink. After the sleeve was shrunk on the cage blank, the pockets were drilled in a conventional manner and a 75-millimeter-bore bearing was run in this reinforced cage for 90 hours at speeds up to 30,000 rpm, which is equivalent to DN value of 2,255,000.

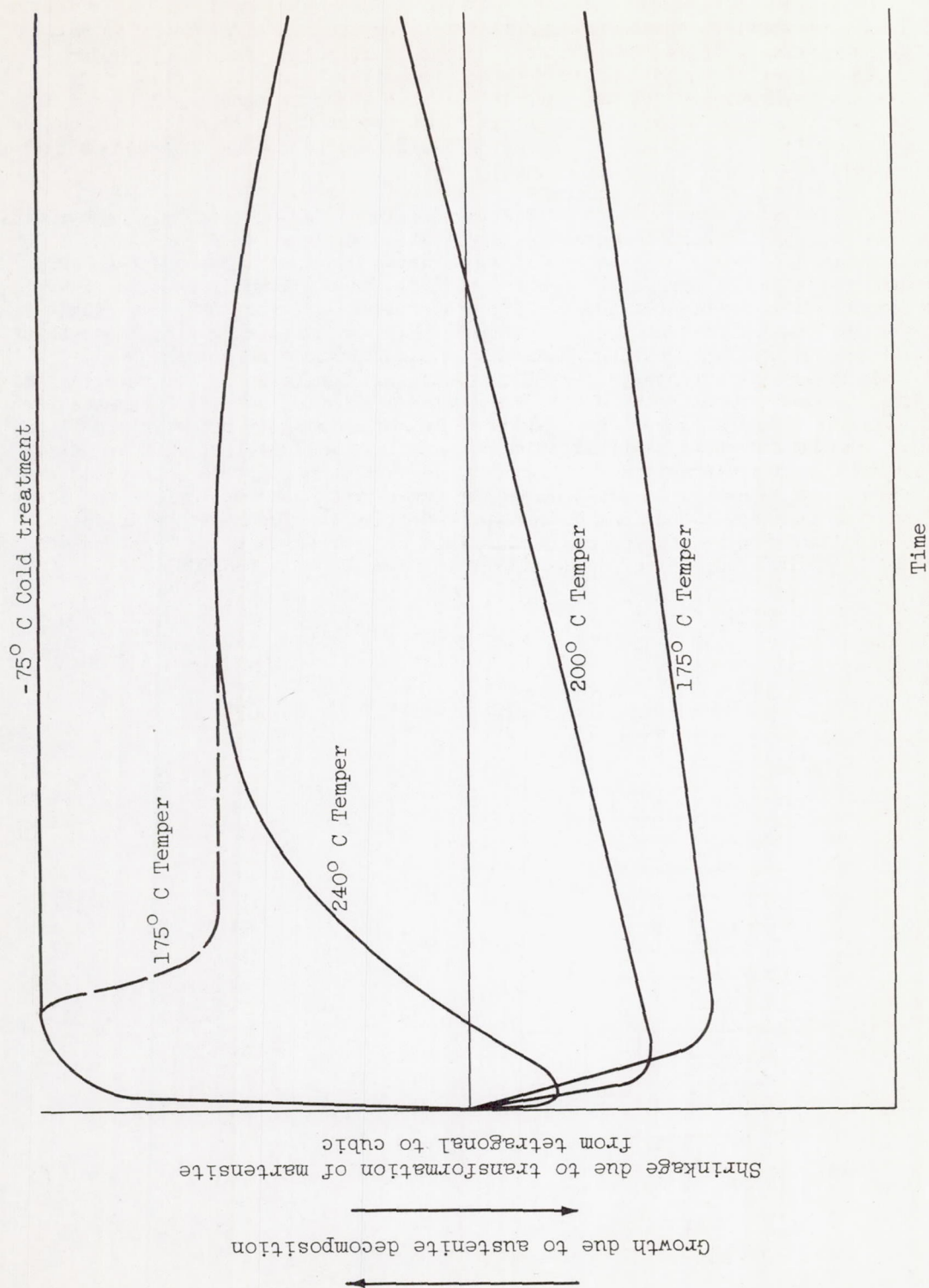


Figure 1. - Effect of temperature and time on growth of a quenched SAE 52100 ring.

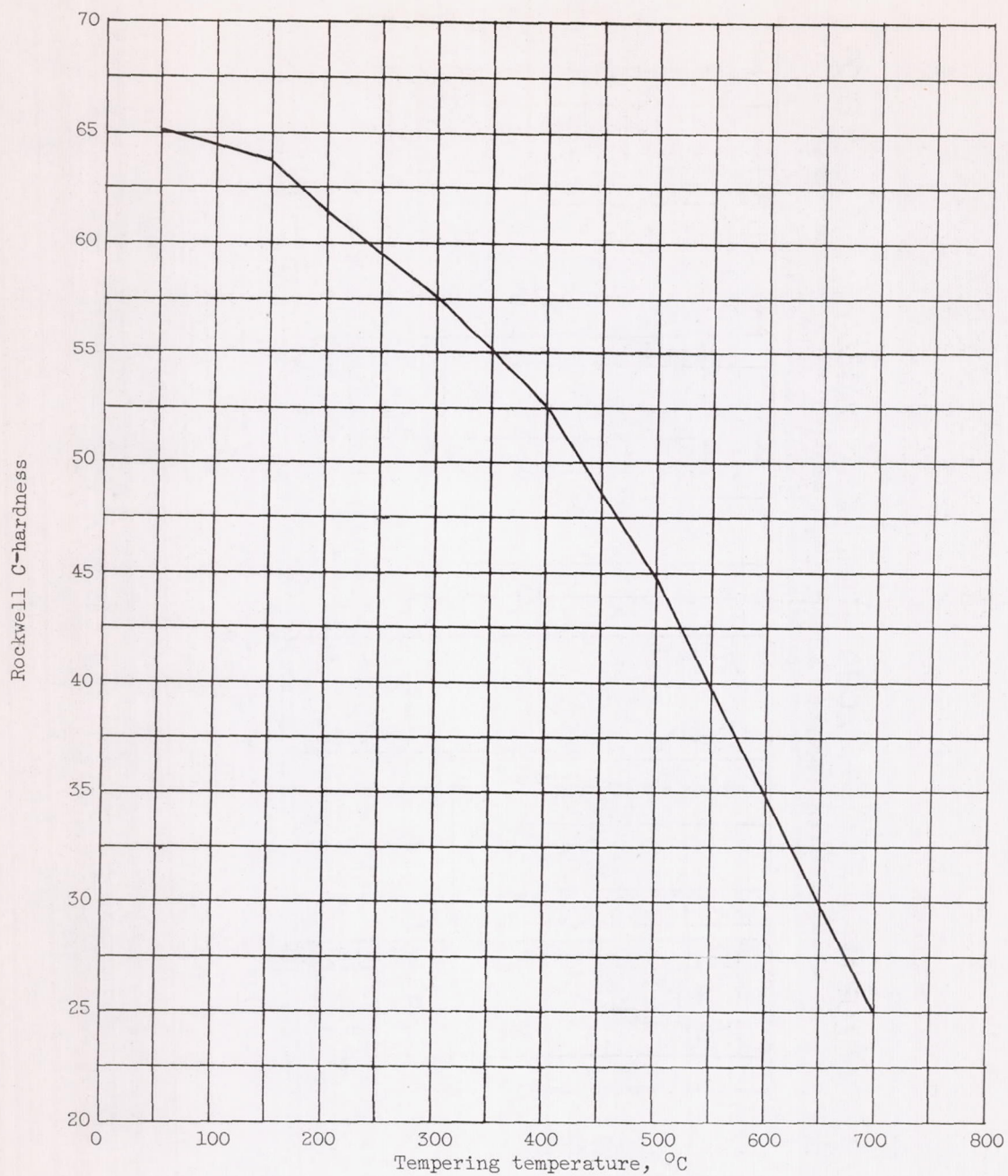


Figure 2. - Hardness against tempering temperature for SAE 52100 oil-quenched from 843° C.

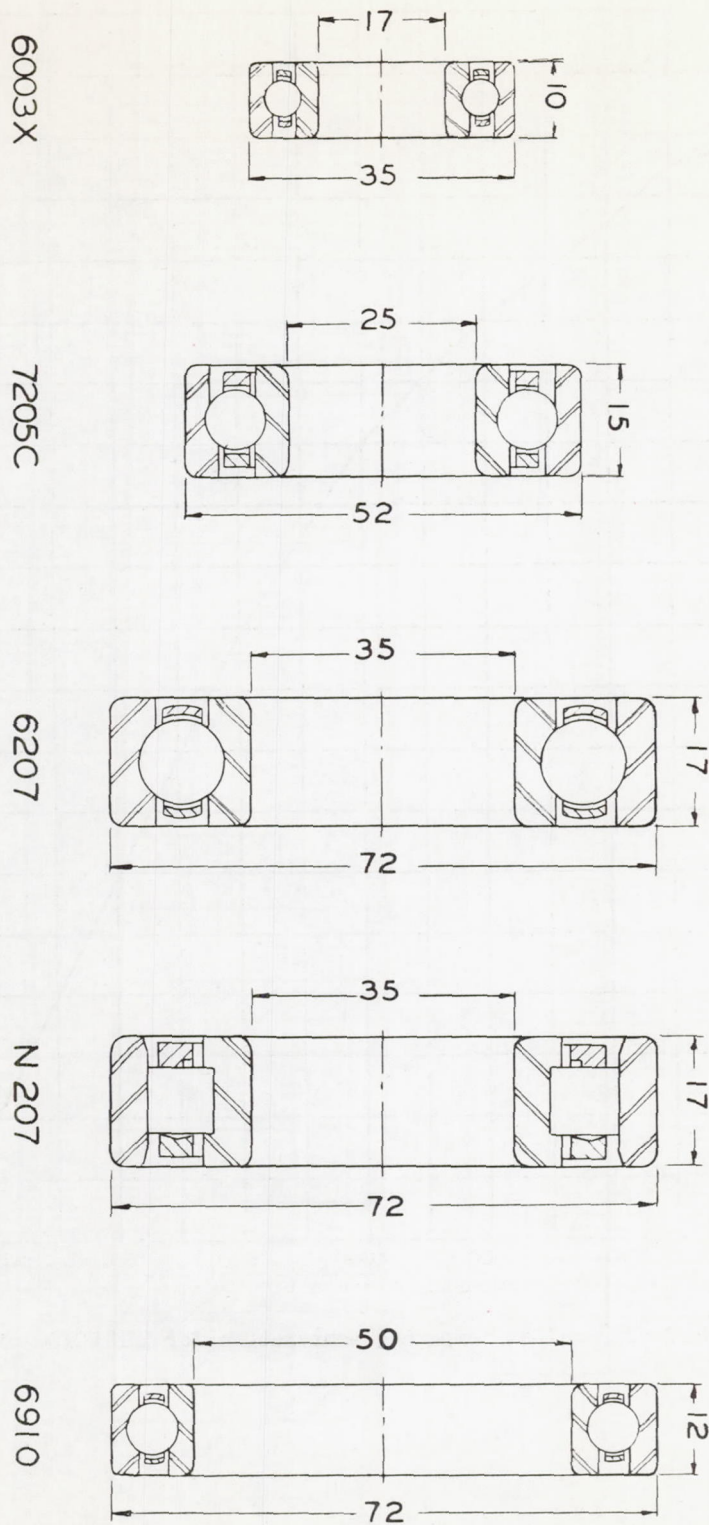
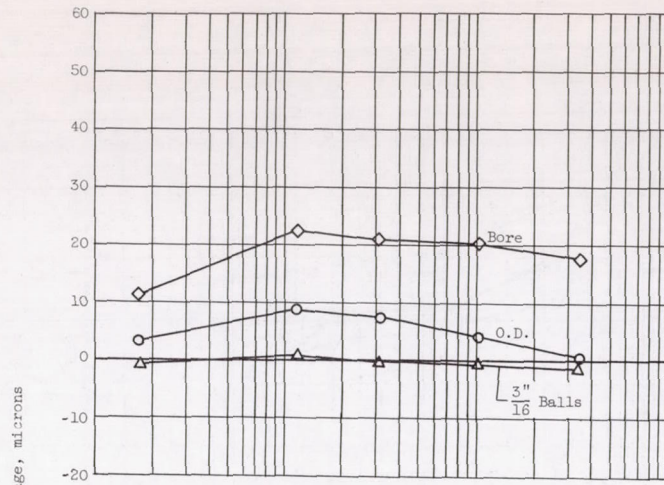
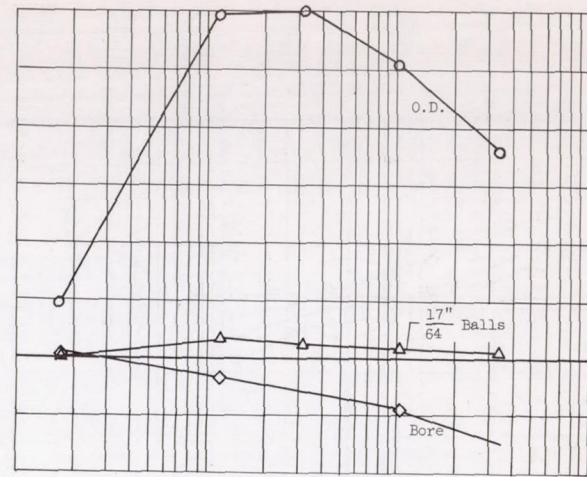


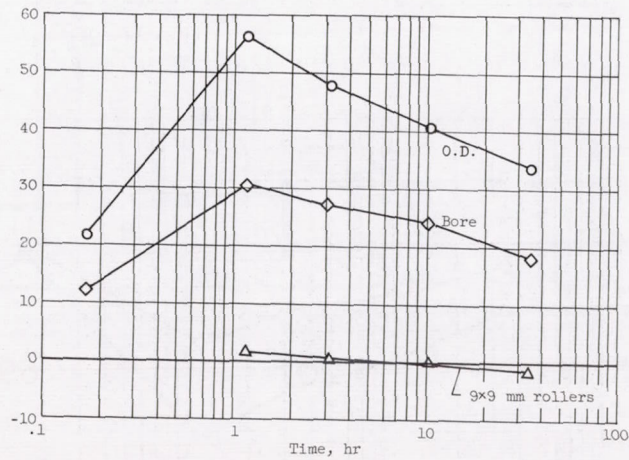
Figure 3. - Typical accessory case bearings.



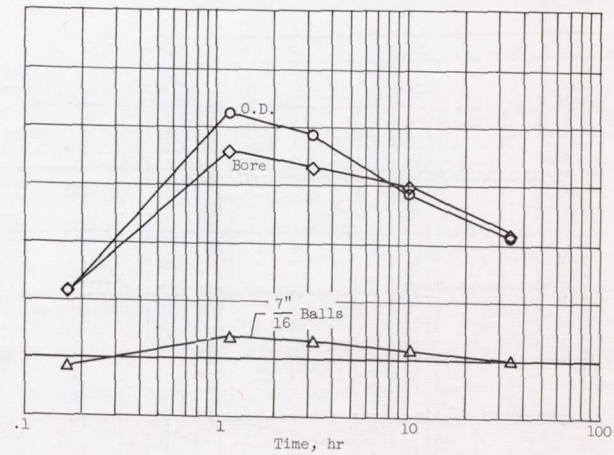
(a) Diameter changes due to tempering at 500° F. Bearing No. 6003XJ.



(b) Diameter changes due to tempering at 500° F. Bearing No. 6910J.

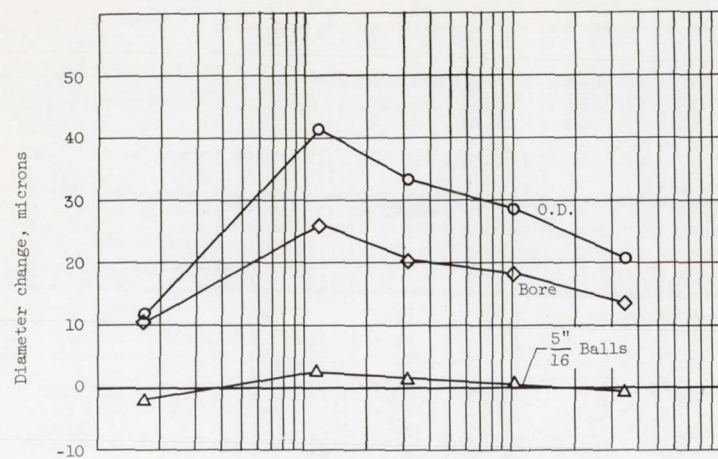


(c) Diameter changes due to tempering at 500° F. Bearing No. N207M.

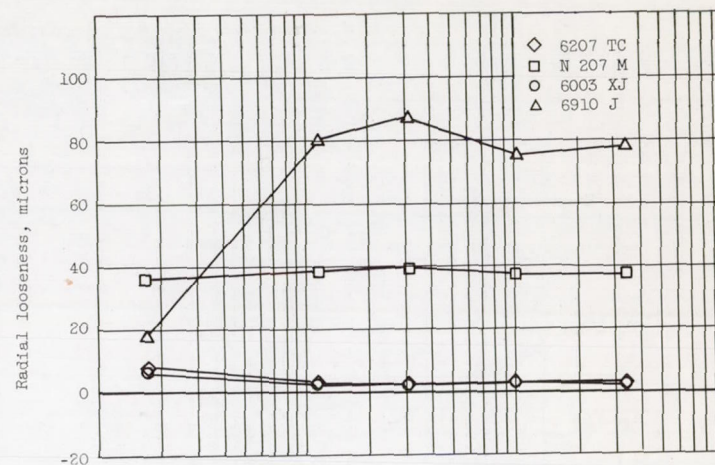


(d) Diameter changes due to tempering at 500° F. Bearing No. 6207 TC.

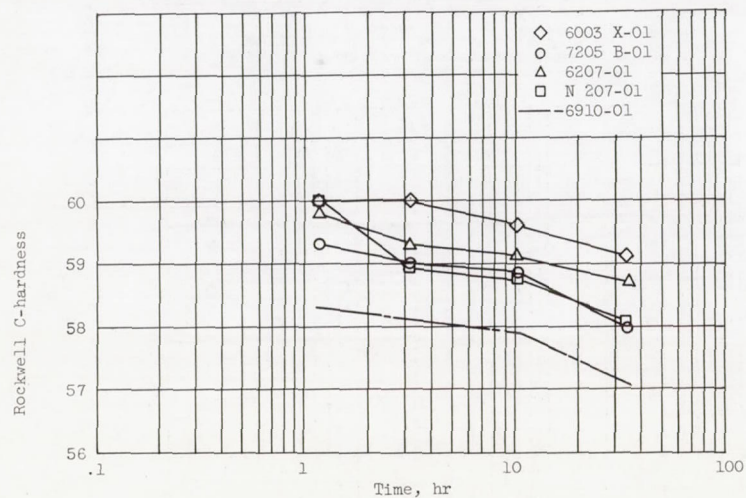
Figure 4. - Dimensional and hardness changes in accessory bearings of figure 3 after various periods of time at 500° F.



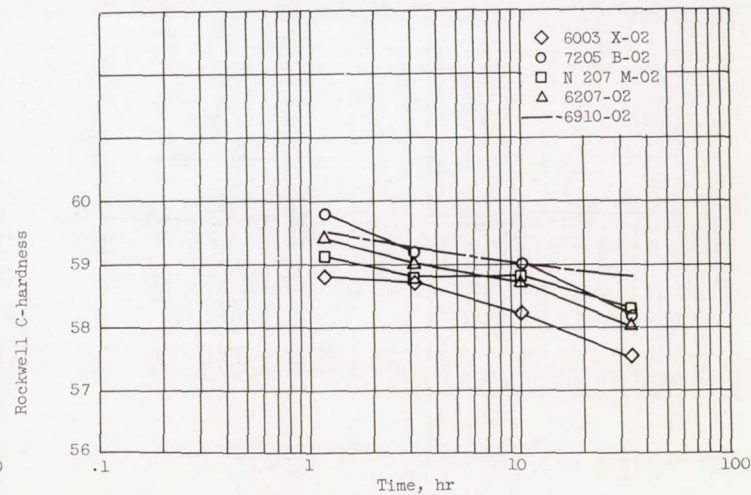
(e) Diameter changes due to tempering at 500° F. Bearing No. 7205/CTC/C78/G2



(f) Radial looseness after tempering at 500° F.



(g) Hardness after tempering at 500° F.



(h) Hardness after tempering at 500° F.

Figure 4. - Concluded. Dimensional and hardness changes in accessory bearings of figure 3 after various periods of time at 500° F.

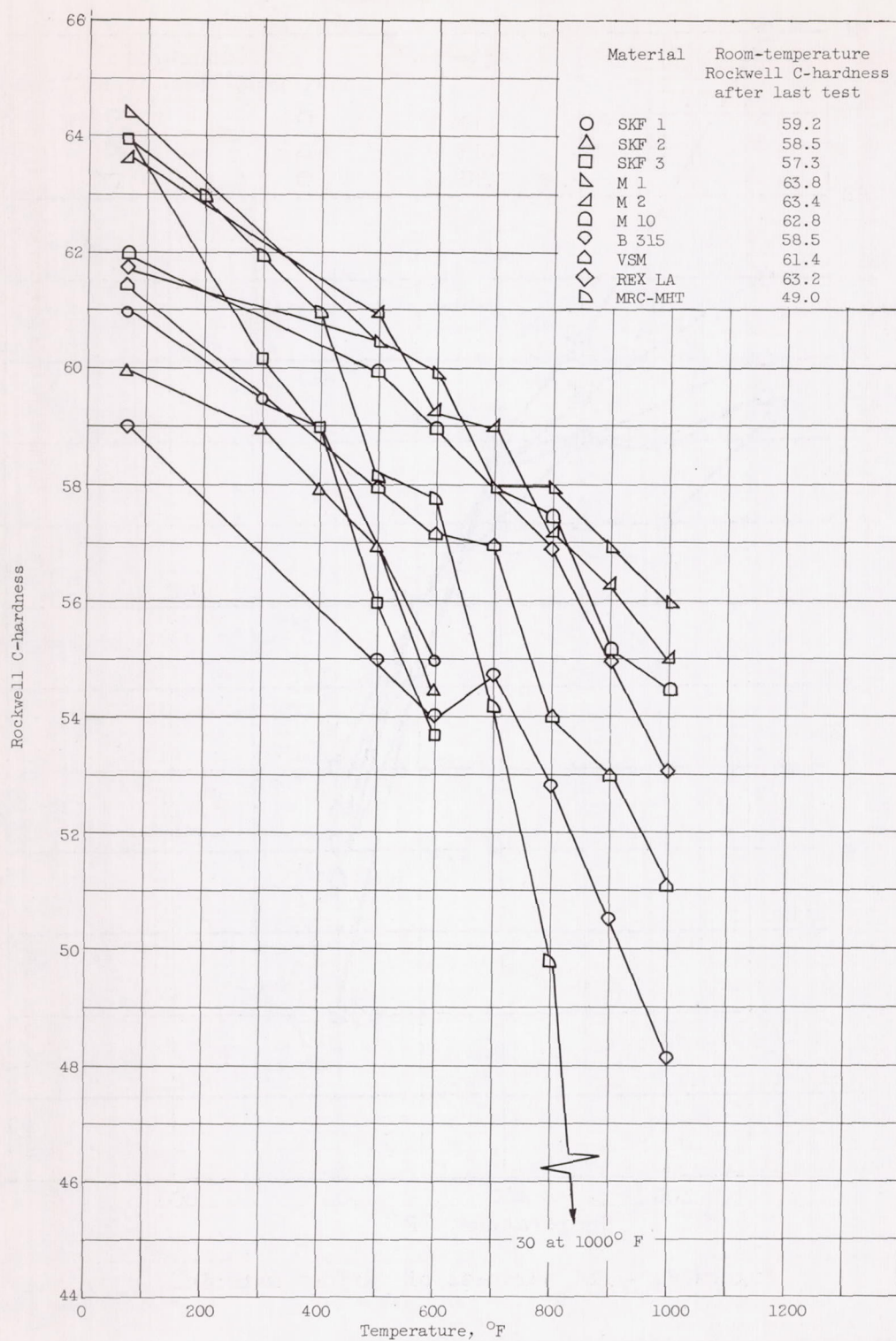


Figure 5. - Hot hardness of various materials. Time at each temperature, 1 hour.

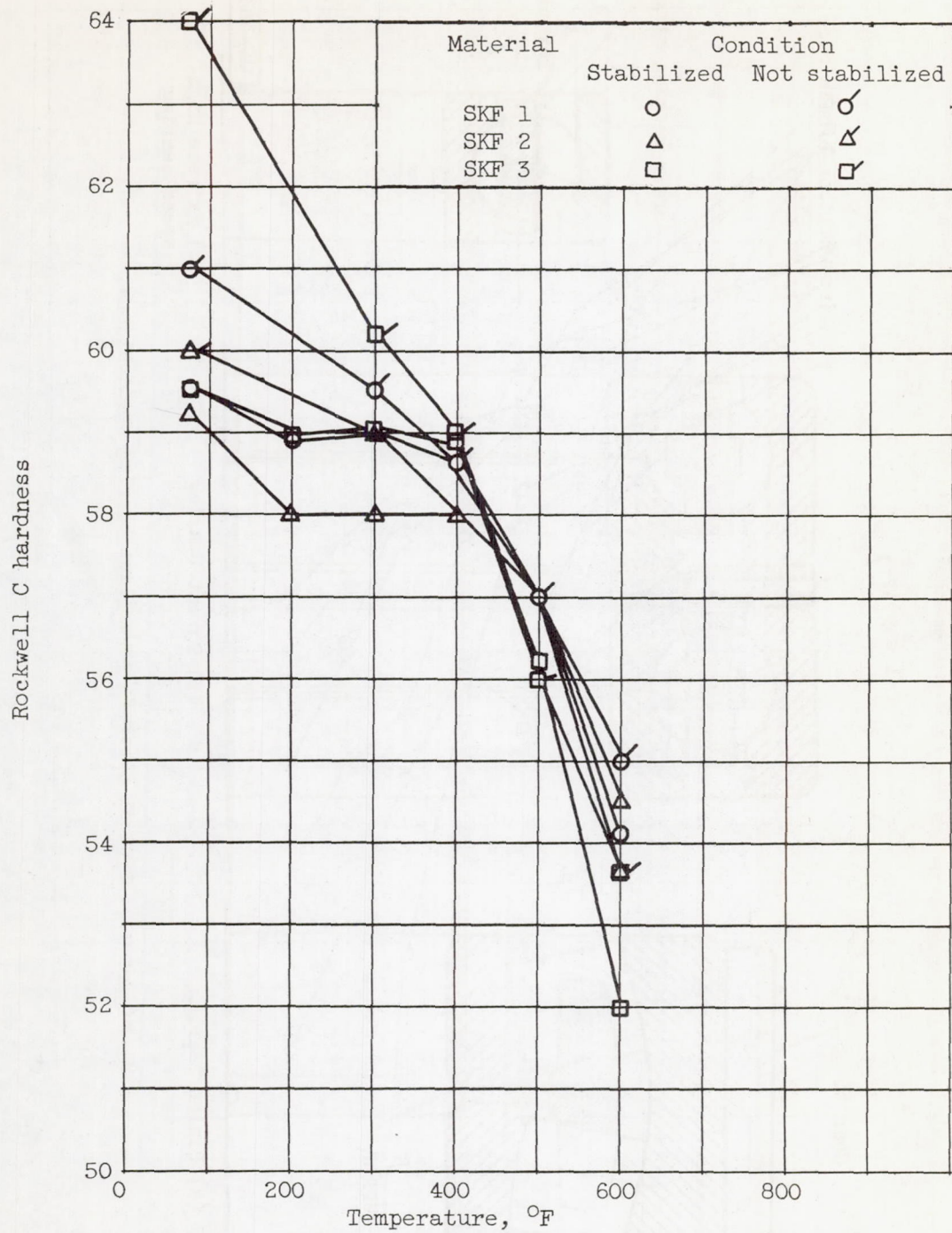


Figure 6. - Hot hardness of various material.

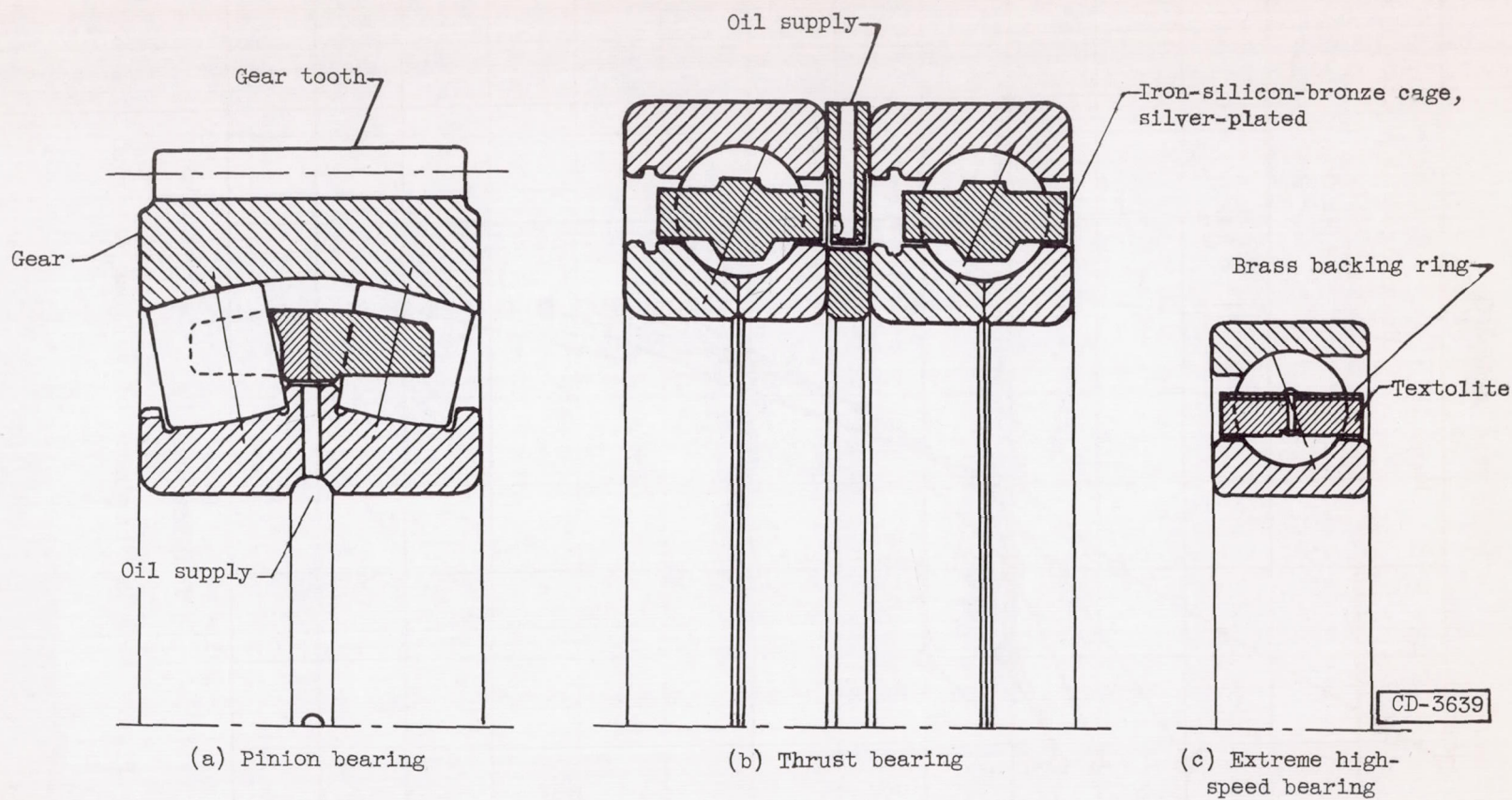


Figure 7. - Typical aircraft powerplant bearing applications.

